

Re Denver Flotation Comparison

Doc

We are particularly anxious to know what the effect of the increased height of liquid in a larger unit will do to the still time, therefore, to change order to the #7 Denver Sub-A Flotation cell which appears to have a volume at least three times that of the unit now being sent to us?

Harry Gold
Joseph E. P. Bowers

57(a)

1997

79

53

10

10-670

1

1

1998, 1999, 2000, 2001, 2002, 2003, 2004, 2005, 2006, 2007, 2008, 2009, 2010, 2011, 2012, 2013, 2014, 2015, 2016, 2017, 2018, 2019, 2020, 2021, 2022, 2023, 2024, 2025, 2026, 2027, 2028, 2029, 2030, 2031, 2032, 2033, 2034, 2035, 2036, 2037, 2038, 2039, 2040, 2041, 2042, 2043, 2044, 2045, 2046, 2047, 2048, 2049, 2050, 2051, 2052, 2053, 2054, 2055, 2056, 2057, 2058, 2059, 2060, 2061, 2062, 2063, 2064, 2065, 2066, 2067, 2068, 2069, 2070, 2071, 2072, 2073, 2074, 2075, 2076, 2077, 2078, 2079, 2080, 2081, 2082, 2083, 2084, 2085, 2086, 2087, 2088, 2089, 2090, 2091, 2092, 2093, 2094, 2095, 2096, 2097, 2098, 2099, 2100, 2101, 2102, 2103, 2104, 2105, 2106, 2107, 2108, 2109, 2110, 2111, 2112, 2113, 2114, 2115, 2116, 2117, 2118, 2119, 2120, 2121, 2122, 2123, 2124, 2125, 2126, 2127, 2128, 2129, 2130, 2131, 2132, 2133, 2134, 2135, 2136, 2137, 2138, 2139, 2140, 2141, 2142, 2143, 2144, 2145, 2146, 2147, 2148, 2149, 2150, 2151, 2152, 2153, 2154, 2155, 2156, 2157, 2158, 2159, 2160, 2161, 2162, 2163, 2164, 2165, 2166, 2167, 2168, 2169, 2170, 2171, 2172, 2173, 2174, 2175, 2176, 2177, 2178, 2179, 2180, 2181, 2182, 2183, 2184, 2185, 2186, 2187, 2188, 2189, 2190, 2191, 2192, 2193, 2194, 2195, 2196, 2197, 2198, 2199, 2200, 2201, 2202, 2203, 2204, 2205, 2206, 2207, 2208, 2209, 2210, 2211, 2212, 2213, 2214, 2215, 2216, 2217, 2218, 2219, 2220, 2221, 2222, 2223, 2224, 2225, 2226, 2227, 2228, 2229, 2230, 2231, 2232, 2233, 2234, 2235, 2236, 2237, 2238, 2239, 2240, 2241, 2242, 2243, 2244, 2245, 2246, 2247, 2248, 2249, 2250, 2251, 2252, 2253, 2254, 2255, 2256, 2257, 2258, 2259, 2260, 2261, 2262, 2263, 2264, 2265, 2266, 2267, 2268, 2269, 2270, 2271, 2272, 2273, 2274, 2275, 2276, 2277, 2278, 2279, 2280, 2281, 2282, 2283, 2284, 2285, 2286, 2287, 2288, 2289, 2290, 2291, 2292, 2293, 2294, 2295, 2296, 2297, 2298, 2299, 2300, 2301, 2302, 2303, 2304, 2305, 2306, 2307, 2308, 2309, 2310, 2311, 2312, 2313, 2314, 2315, 2316, 2317, 2318, 2319, 2320, 2321, 2322, 2323, 2324, 2325, 2326, 2327, 2328, 2329, 2330, 2331, 2332, 2333, 2334, 2335, 2336, 2337, 2338, 2339, 2340, 2341, 2342, 2343, 2344, 2345, 2346, 2347, 2348, 2349, 2350, 2351, 2352, 2353, 2354, 2355, 2356, 2357, 2358, 2359, 2360, 2361, 2362, 2363, 2364, 2365, 2366, 2367, 2368, 2369, 2370, 2371, 2372, 2373, 2374, 2375, 2376, 2377, 2378, 2379, 2380, 2381, 2382, 2383, 2384, 2385, 2386, 2387, 2388, 2389, 2390, 2391, 2392, 2393, 2394, 2395, 2396, 2397, 2398, 2399, 2400, 2401, 2402, 2403, 2404, 2405, 2406, 2407, 2408, 2409, 2410, 2411, 2412, 2413, 2414, 2415, 2416, 2417, 2418, 2419, 2420, 2421, 2422, 2423, 2424, 2425, 2426, 2427, 2428, 2429, 2430, 2431, 2432, 2433, 2434, 2435, 2436, 2437, 2438, 2439, 2440, 2441, 2442, 2443, 2444, 2445, 2446, 2447, 2448, 2449, 2450, 2451, 2452, 2453, 2454, 2455, 2456, 2457, 2458, 2459, 2460, 2461, 2462, 2463, 2464, 2465, 2466, 2467, 2468, 2469, 2470, 2471, 2472, 2473, 2474, 2475, 2476, 2477, 2478, 2479, 2480, 2481, 2482, 2483, 2484, 2485, 2486, 2487, 2488, 2489, 2490, 2491, 2492, 2493, 2494, 2495, 2496, 2497, 2498, 2499, 2500, 2501, 2502, 2503, 2504, 2505, 2506, 2507, 2508, 2509, 2510, 2511, 2512, 2513, 2514, 2515, 2516, 2517, 2518, 2519, 2520, 2521, 2522, 2523, 2524, 2525, 2526, 2527, 2528, 2529, 2530, 2531, 2532, 2533, 2534, 2535, 2536, 2537, 2538, 2539, 2540, 2541, 2542, 2543, 2544, 2545, 2546, 2547, 2548, 2549, 2550, 2551, 2552, 2553, 2554, 2555, 2556, 2557, 2558, 2559, 2560, 2561, 2562, 2563, 2564, 2565, 2566, 2567, 2568, 2569, 2570, 2571, 2572, 2573, 2574, 2575, 2576, 2577, 2578, 2579, 2580, 2581, 2582, 2583, 2584, 2585, 2586, 2587, 2588, 2589, 2590, 2591, 2592, 2593, 2594, 2595, 2596, 2597, 2598, 2599, 2600, 2601, 2602, 2603, 2604, 2605, 2606, 2607, 2608, 2609, 2610, 2611, 2612, 2613, 2614, 2615, 2616, 2617, 2618, 2619, 2620, 2621, 2622, 2623, 2624, 2625, 2626, 2627, 2628, 2629, 2630, 2631, 2632, 2633, 2634, 2635, 2636, 2637, 2638, 2639, 2640, 2641, 2642, 2643, 2644, 2645, 2646, 2647, 2648, 2649, 2650, 2651, 2652, 2653, 2654, 2655, 2656, 2657, 2658, 2659, 2660, 2661, 2662, 2663, 2664, 2665, 2666, 2667, 2668, 2669, 2670, 2671, 2672, 2673, 2674, 2675, 2676, 2677, 2678, 2679, 26

11/11/71

7900 mi
over ACTA
1000 mi (condensed atmosphere)

ACTA

198

5070

1970

1970

1970

1970

1970

1970

1970

Page 14

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average bill

average bill

the co. can. / th.

○ per cor / per edn.

apparent. above

explaning.

note of the above.

the / per edn / th.

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—

o.s.

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(7)

49.3

3.4

17.7

54.670

9.0

49.3

3.4

17.7

46.390

11.4

24.5

1.00

1.00

9.000

1.00

4.44

8.5

= 1.69

1.69

0.84

6.7



11.4

3.0

7.4

16.8

1.7

0.6

0.4

1.2

0.1

0.8

7 (12.2)

1.7

Program

Finish analytical methods
for Furnace leachings - June 2

2. Meanwhile, get ready for run
on decomposition of NaHCO_3 , due
to determine
a. Rate of evolution of CO_2
b. Steam used.
make run - June 2

3. Begin work on separation of salt
from Furnace leachings after
preliminary tests, try out at least
one scheme - July

Leached + CO_2 -

May 2

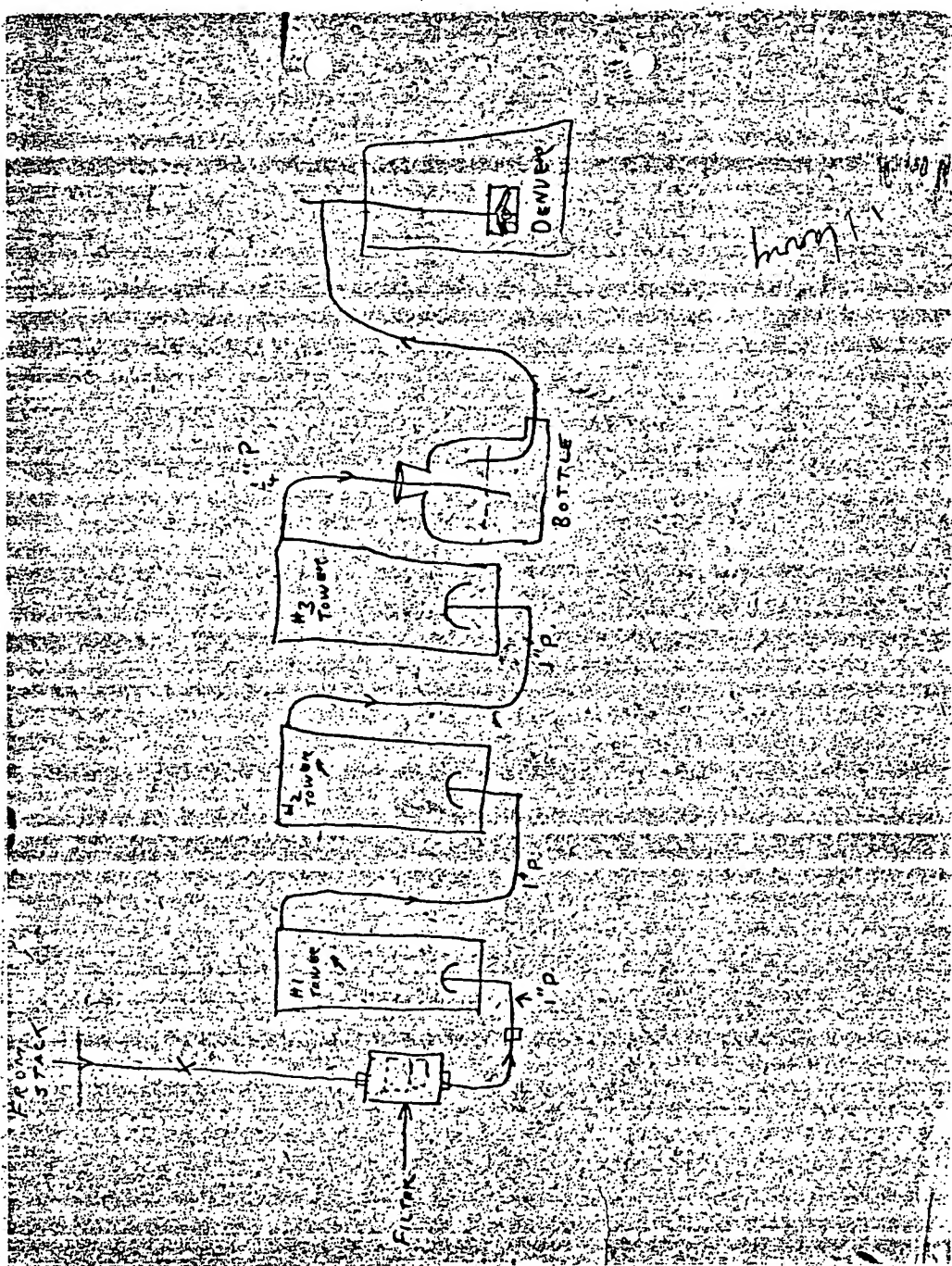
Program

1. Try absorption on larger scale unit to determine:

- a. power cost
- b. method of operation
- c. efficiency in removing CO_2

2. measure steam necessary for decomposition. This can be done on a 2 or 3 gal scale

3. study removal of SO_2 and dust



hwy 1

5/29/52

Doc

The O₂ analyzer equipment is drawing
fuel gas through three columns and
into the absorber proper; there is
apparently no decrease in the
rate of flow.

[illegible]

Time	Temp	Wind	Speed	Gen. Avg. (Yr. 1951-55)		Precip.	T. Fraction (M. of N. H. 50)	
				5 x 5 (0.5 mi)	5 x 5 (0.5 mi)		5 x 5 (0.5 mi)	5 x 5 (0.5 mi)
1	10.5	0.0	12.10	11.2	0.0	47	24.2	48.9
2	11.5	0.0	12.10	11.2	0.0	49	22.8	47.3
3	12.5	0.0	12.10	11.2	0.0	48	22.6	47.6
4	13.5	0.0	12.10	11.2	0.0	49	22.8	47.3
5	14.5	0.0	12.10	11.2	0.0	49	22.8	47.3
6	15.5	0.0	12.10	11.2	0.0	49	22.8	47.3
7	16.5	0.0	12.10	11.2	0.0	49	22.8	47.3
8	17.5	0.0	12.10	11.2	0.0	49	22.8	47.3
9	18.5	0.0	12.10	11.2	0.0	49	22.8	47.3
10	19.5	0.0	12.10	11.2	0.0	49	22.8	47.3
11	20.5	0.0	12.10	11.2	0.0	49	22.8	47.3
12	21.5	0.0	12.10	11.2	0.0	49	22.8	47.3
13	22.5	0.0	12.10	11.2	0.0	49	22.8	47.3
14	23.5	0.0	12.10	11.2	0.0	49	22.8	47.3
15	24.5	0.0	12.10	11.2	0.0	49	22.8	47.3
16	25.5	0.0	12.10	11.2	0.0	49	22.8	47.3
17	26.5	0.0	12.10	11.2	0.0	49	22.8	47.3
18	27.5	0.0	12.10	11.2	0.0	49	22.8	47.3
19	28.5	0.0	12.10	11.2	0.0	49	22.8	47.3
20	29.5	0.0	12.10	11.2	0.0	49	22.8	47.3
21	30.5	0.0	12.10	11.2	0.0	49	22.8	47.3
22	31.5	0.0	12.10	11.2	0.0	49	22.8	47.3
23	32.5	0.0	12.10	11.2	0.0	49	22.8	47.3
24	33.5	0.0	12.10	11.2	0.0	49	22.8	47.3
25	34.5	0.0	12.10	11.2	0.0	49	22.8	47.3
26	35.5	0.0	12.10	11.2	0.0	49	22.8	47.3
27	36.5	0.0	12.10	11.2	0.0	49	22.8	47.3
28	37.5	0.0	12.10	11.2	0.0	49	22.8	47.3
29	38.5	0.0	12.10	11.2	0.0	49	22.8	47.3
30	39.5	0.0	12.10	11.2	0.0	49	22.8	47.3
31	40.5	0.0	12.10	11.2	0.0	49	22.8	47.3
32	41.5	0.0	12.10	11.2	0.0	49	22.8	47.3
33	42.5	0.0	12.10	11.2	0.0	49	22.8	47.3
34	43.5	0.0	12.10	11.2	0.0	49	22.8	47.3
35	44.5	0.0	12.10	11.2	0.0	49	22.8	47.3
36	45.5	0.0	12.10	11.2	0.0	49	22.8	47.3
37	46.5	0.0	12.10	11.2	0.0	49	22.8	47.3
38	47.5	0.0	12.10	11.2	0.0	49	22.8	47.3
39	48.5	0.0	12.10	11.2	0.0	49	22.8	47.3
40	49.5	0.0	12.10	11.2	0.0	49	22.8	47.3
41	50.5	0.0	12.10	11.2	0.0	49	22.8	47.3
42	51.5	0.0	12.10	11.2	0.0	49	22.8	47.3
43	52.5	0.0	12.10	11.2	0.0	49	22.8	47.3
44	53.5	0.0	12.10	11.2	0.0	49	22.8	47.3
45	54.5	0.0	12.10	11.2	0.0	49	22.8	47.3
46	55.5	0.0	12.10	11.2	0.0	49	22.8	47.3
47	56.5	0.0	12.10	11.2	0.0	49	22.8	47.3
48	57.5	0.0	12.10	11.2	0.0	49	22.8	47.3
49	58.5	0.0	12.10	11.2	0.0	49	22.8	47.3
50	59.5	0.0	12.10	11.2	0.0	49	22.8	47.3

20. On most of the above, the following comments are made:

② 1859 5th Dec

7 41.50 / 1.50

1.22 41.50

1.22 41.50

0.64

no profit

1.22 41.50 / 1.50

1.00 →

1.00 41.50

~~1.00 41.50~~

50

5' con from 10' ft

17' ft

7' ft per 100

122' ft per 100

122,000 per hr

700' also 4/1

17

1117

1 absorber 700 gals

2 intake section

Cap

Absorber 50' ft p/h

Cap 2.5 gals

Large

950 gals

50.00

300' Times

10

15000' ft p/h

10' Time plant

10 units

50 Hc

13 liter unit

11 liter unit

7.5 gal unit

5' H₂O section

10 1/2" Hg = 140" H₂O

1 gal = 20' ft

2.5 gals cap

50' ft

950

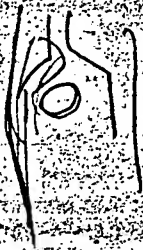
15,000' ft

10

1 gal = 20' ft p/h

75

15,000' ft



1. 0.7% CO₂ in air
 2. 4.0% CO₂ in air
 3. 10.0% CO₂ in air
 4. 20.0% CO₂ in air
 5. 30.0% CO₂ in air
 6. 40.0% CO₂ in air
 7. 50.0% CO₂ in air
 8. 60.0% CO₂ in air
 9. 70.0% CO₂ in air
 10. 80.0% CO₂ in air
 11. 90.0% CO₂ in air
 12. 100.0% CO₂ in air

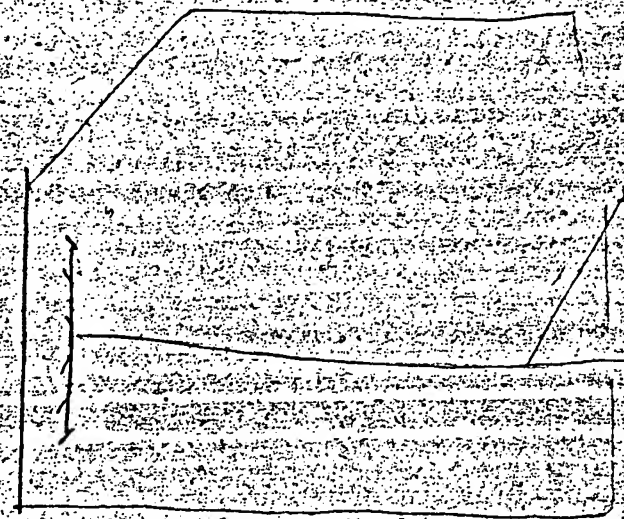
1. 0.7% CO₂ in air
 2. 4.0% CO₂ in air
 3. 10.0% CO₂ in air
 4. 20.0% CO₂ in air
 5. 30.0% CO₂ in air
 6. 40.0% CO₂ in air
 7. 50.0% CO₂ in air
 8. 60.0% CO₂ in air
 9. 70.0% CO₂ in air
 10. 80.0% CO₂ in air
 11. 90.0% CO₂ in air
 12. 100.0% CO₂ in air

0.2

1. 0.7% CO₂ in air
2. 4.0% CO₂ in air
3. 10.0% CO₂ in air
4. 20.0% CO₂ in air
5. 30.0% CO₂ in air
6. 40.0% CO₂ in air
7. 50.0% CO₂ in air
8. 60.0% CO₂ in air
9. 70.0% CO₂ in air
10. 80.0% CO₂ in air
11. 90.0% CO₂ in air
12. 100.0% CO₂ in air

75

1. 0.7% CO₂ in air
 2. 4.0% CO₂ in air
 3. 10.0% CO₂ in air
 4. 20.0% CO₂ in air
 5. 30.0% CO₂ in air
 6. 40.0% CO₂ in air
 7. 50.0% CO₂ in air
 8. 60.0% CO₂ in air
 9. 70.0% CO₂ in air
 10. 80.0% CO₂ in air
 11. 90.0% CO₂ in air
 12. 100.0% CO₂ in air



1. 0.7% CO₂ in air
 2. 4.0% CO₂ in air
 3. 10.0% CO₂ in air
 4. 20.0% CO₂ in air
 5. 30.0% CO₂ in air
 6. 40.0% CO₂ in air
 7. 50.0% CO₂ in air
 8. 60.0% CO₂ in air
 9. 70.0% CO₂ in air
 10. 80.0% CO₂ in air
 11. 90.0% CO₂ in air
 12. 100.0% CO₂ in air

scale toward this and the
following should be ~~done~~ carried out

1. Try the alternator in a 750
gal unit — either at Penn
or here

2. ~~Try a~~ 750 gal unit (couple
with a 1 h.p. motor) & try
the alternator here

Reconstruction

Reconstruction
of the
Creek

75

100

100

() () ()

$$102,000 \text{ ft}^3/\text{h} \times 2.55 = 260,600 \text{ gal/h}$$

$$\frac{102,000}{2.55} \times 1.5 = 5,000 \text{ gal/h}$$

$$\frac{50}{2} = 25$$
$$= \frac{6.8 \text{ units}}{1 \text{ unit}}$$

46.8
24.6

46.8
12.2

=

26.090

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8
24.6

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

46.8

24.2

6.4 am - Nucleus

1.8 am - Nucleus

$$118 - 4.0 = 7.8$$

$$0.758 \times 2.2 = 0.644 \text{ m.c. / h}$$

$$2.5 \times 2.2 = 2.2 \text{ gals}$$

m.c. / h

$$2.2 \times 2.2 = 4.84$$

$$10,000 \div 4.84 = 2066.1157$$

$$2.2 \times 2.2 = 4.84$$

$$2.2 \times 2.2 = 4.84$$

$$750$$

$$0.644 \times 2.2 = 1.4168$$

5 units

4,000
1,100

4,500 x 5

→ 12,500 cost for abn

~~17,000~~
2,000

Settler

5,000

peasants

Scrubber

5,000

~~Scrubber~~

instruments

3,000

Heat Exch

2,000

Pig Pumps

1,000

31,000

Gas flow

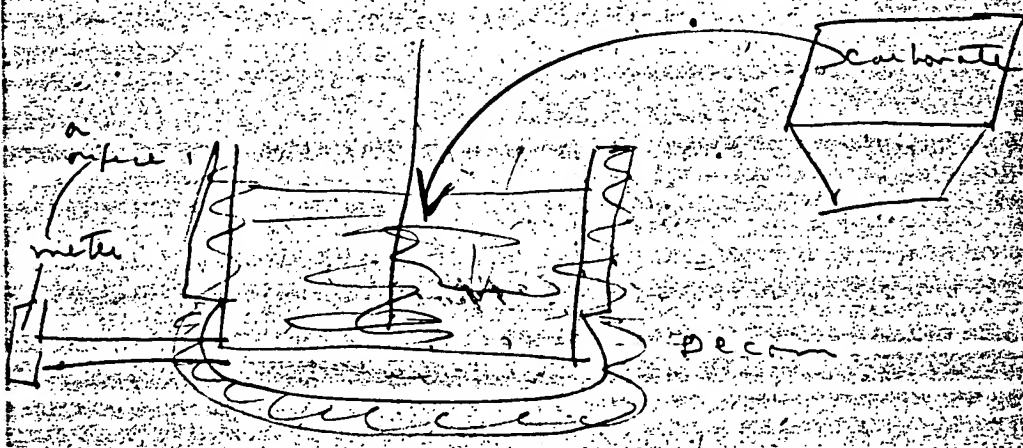
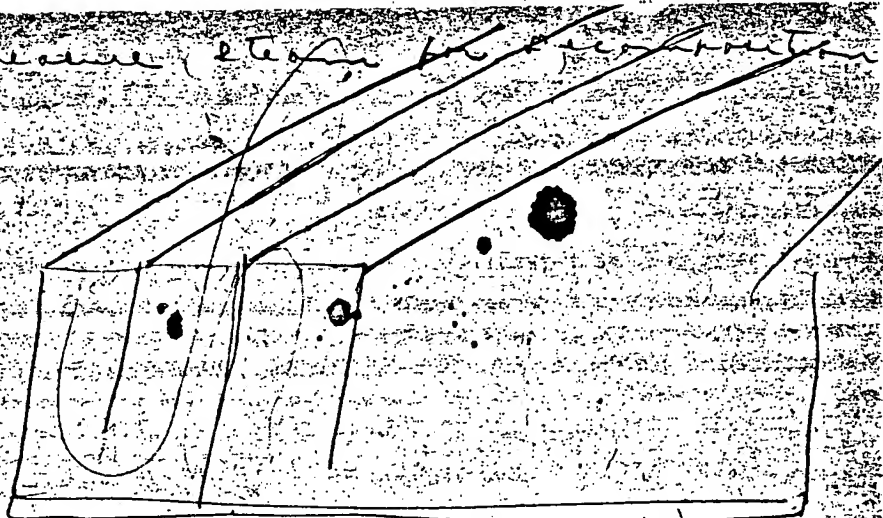
Gas ~~flow~~ meters

500 meters

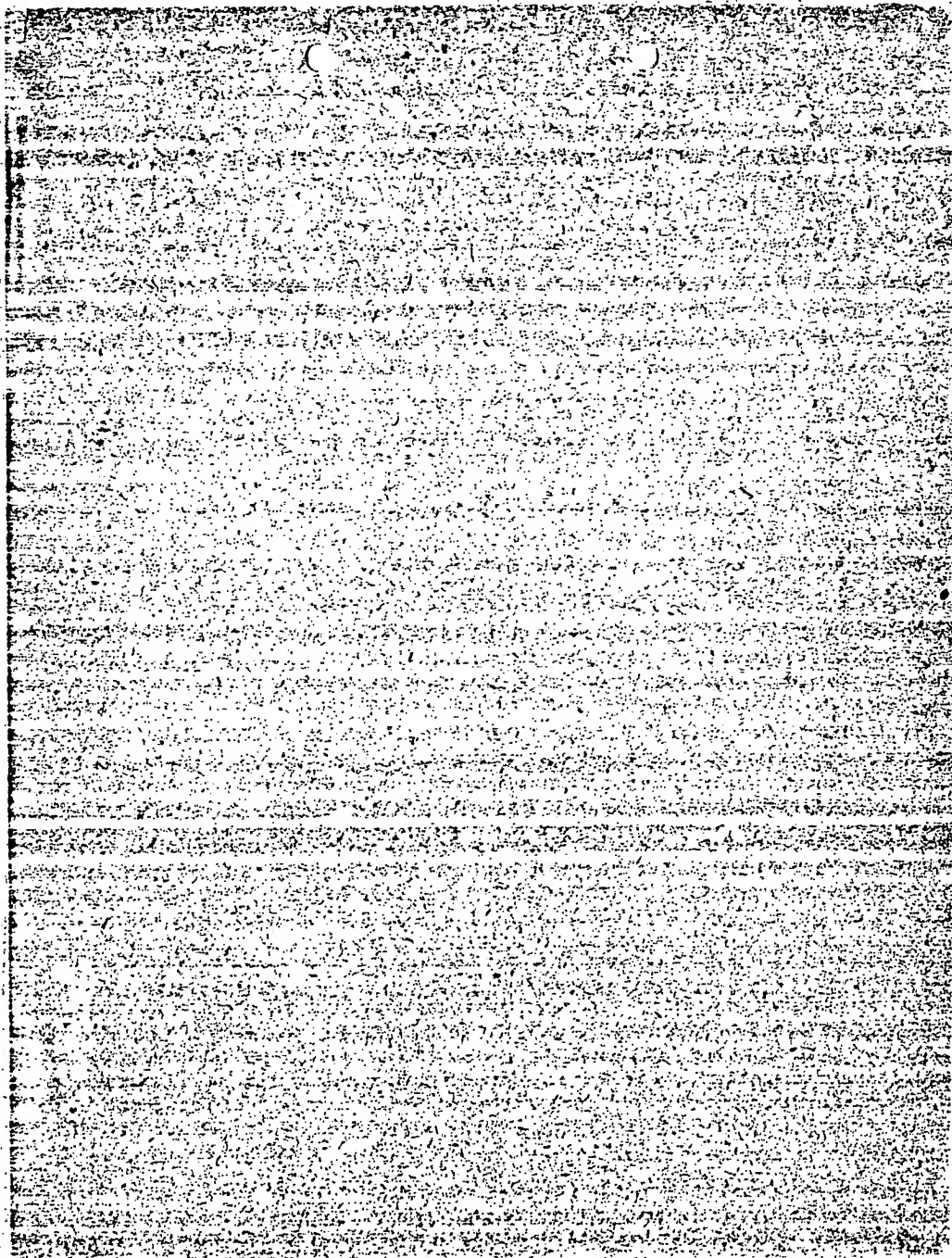
Steam meters

Water meters

measure (2 to 3 ft) per section



on 15#



R U N 43

4/30/41.

Data Sample No.	Time	Gas Rate C.F.P.M.	Speed R.P.M.	Gas Analysis % CO ₂			Temp °C	Titration ml. of N H ₂ SO ₄			
				Inlet	Krit orsat	Corrected Krit		Solution		Total	
1	1:45	0.50 (1)	2050	11.0	5.8		46				
2	2:00	0.70 (2)	2050	11.0	5.8		47				
3	2:15	0.60 (3)	2050	11.6	6.2		50				
4	2:30	0.60 (4)	2040	11.6	6.0		51				
5	2:45	0.60 (5)	2100	11.0	6.2		51				
6	3:00	0.60 (6)	2100	11.0	6.0		50				
7 (9)	3:30	0.90 (7)	2270 (11)	11.0	8.0		49				
8 (10)	4:30	0.75 (8)	2250	{ 8 } 5.2	1.6 (?)		50				

Notes:-

- (1) - 8 liters
- (8), (5), (4), (5), (6), (7), (8), - 10 liters.
- (9) - Inlet Valve wide open.
- (10) - No pump, just Denver station.
- (11) - Suction = 10.5" Hg.

Continued

Results:-

1. By the use of standpipes, 10 liters were kept in the absorber. This was done even with a gas rate as high as 54 ft. ³/hr.
2. The rotor speed was too low (2050 r.p.m. as compared with previous values of 2300 to 2400 r.p.m.) This was thought due to the partial closing of the inlet valve in regulating the gas flow.
3. With the above conditions an average inlet gas of 11.0% CO₂ and an exit of 6.0% CO₂ were obtained. The gas rate was 38 ft. ³/hr.
4. The suction pulled by the Denver was found to be 10.5" Hg. and, therefore, it was decided to run the absorber just by the use of this suction. The data were:

a. Speed 22.50 r.p.m.

b. Gas Rate 45 ft. ³/hr.

c. Gas analyses } inlet 12.0
% CO₂ } exit 3.2 (1.6?)

Signed:

Witnesses

R. J. Baird

M. C. Dougherty

Harry Gold
Joseph E. P. Bower

11
of the Omen Plot on
an
admission
announcing

suppose

[illegible]

The same method was used for the determination of the relative mass loss, all measurements of the relative mass loss at 45-48°C

	1	2	3	4	5	6	7
Standard	1	2	3	4	5	6	7
Time	11.15	2.15	2.45				
Height	12.6	12.0	12.0				
Weight	70.0	7.6	4.4				
Amplitude	0.21	0.20	0.15				
Temp	35	41	46				
0.6	2.2	2.14	20.6	12.9	70		
4.0	46.7	46.9	47.1				
1.0			10.12				
2.0			7.80				
3.0			8.90				
4.0			11.90				

The above adaptation may be due to the fact
 that the agitation did not seem as vigorous
 as before. This could possibly have been
 caused by the colored matter being on the
 surface and the surface of the solution and the

1000000

1000000

1000000

1000000

1000000

1000000

1000000

1000000

2

1.0

0.12

0.005

9.1

4.0

2.9

3.0

3

0.5

0.21

0.013

7.6

3.7

4.4

8.3

4

0.6

0.15

10.0

6.3

2

5.1

9

Sample No.	Time	C/P.M.	Speed	Gas Analysis, Vol. %		Temp. °C.	Temperature, m. l. of N. H.C. 5.0			
				Inlet	Outlet		Calibration		Factor	Value
1	11:00	0.80	2170	10.0	4.6	48	244	490	1.01	48.8
2	11:00	0.70	2200	11.6	5.2	48	225	485	1.01	48.8
3	11:30	0.40	2200	11.6	0.6	47	21.6	48.6	1.01	48.8
4	11:45	0.40	2250	10.2	4.6	49	21.4	48.6	1.01	48.8
5	11:55	0.40	2240	10.4	5.4	50	20.5	48.4	1.01	48.8
6	12:05	0.40	2240	11.4	5.8	49	19.7	48.9	1.01	48.8
7	12:10	0.40	2170	11.4	5.0	50	18.9	48.8	1.01	48.8
8	12:15	0.40	2230	11.4	5.8	51	18.5	47.8	1.01	48.8
9	12:20	0.40	2230	11.4	5.8	51	19.1	49.0	1.01	48.8
9(a)	12:25	0.40	2230	11.4	5.8	51	19.1	49.0	1.01	48.8

3-2-3

No.	Time	C.F.P.M.	F.P.M.	Gas Analysis		Temp. °C	T. Reaction		T. of air	
				Inlet	Exit		Vol.	Wt.	Vol.	Wt.
1	1.15	0.50	2050	11.0	3.8	46				
2	2.00	0.70	2059	11.0	5.2	47				
3	2.15	0.60	2050	11.6	6.2	50				
4	2.18	0.60	2040	11.6	6.0	51				
5	2.45	0.40	2100	11.0	6.2	51				
6	2.10	0.60	2100	11.0	6.0	50				
7	3.10	0.90	2270	11.0	8.0	49				
8	4.10	0.75	2250	12.076	11.67	50				

Notes

10-11535-1-1

⑤ Inlet Valve wide open

③ The following are the names of the persons who have been convicted of crimes involving moral turpitude:

Results

For the run of 20 and 25 pps, 10 liter water was in the absorber. This was done with a gas rate as high as $54 \text{ ft}^3/\text{hr}$.

The rotor speed was 100 rpm (2050 rpm as compared with previous values of 2500 or 2400 rpm). This was done due to partial closing of the inlet valve in regulating the gas flow.

With the above conditions an average molar gas of 11.079 CO_2 and an exit of 6.0 CO_2 were obtained. The gas rate was $36 \text{ ft}^3/\text{hr}$.

The reaction pulled by the O-mixer was found to be 10.5 "H_2 and 1.5 "H₂ at the absorber. It was decided to run the absorber just by the

all of this information is to be used

at 2250-2900

at 45 ft 3/4 in.

at 1200

at 32 (11.2)

with these

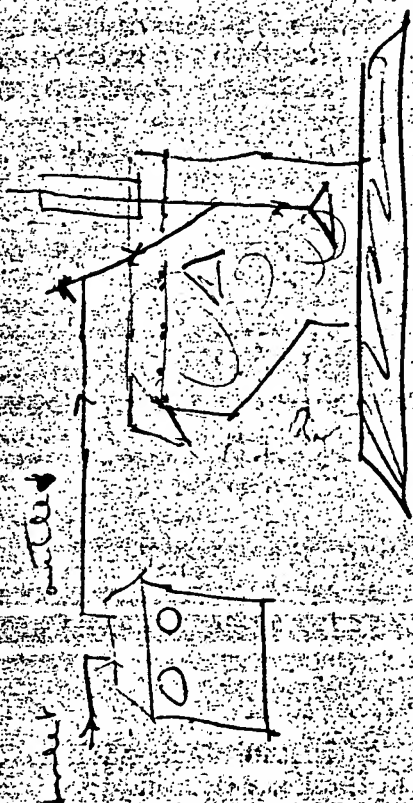
signed:

Henry Gold
Joseph E. Brown

M. E. Dougherty
H. B. Bauld

Run 46

Sample No.	Time	Gas Rate C.F.P.M.	Speed R.P.M.	Gas Analysis, %		Temp. °C.	Titration, ml. of N.H.S.		
				Inlet	Exhaust (orad)		Solution	M.O.	T. & g.
1	1.30						19.8	46.2	
2	2.00	0.20	1520	12.2	2.4	49	17.9	46.0	
3	2.30	0.21	1560	12.3	3.9	49	16.6	45.8	
4	3.00	0.21	1550	12.0	5.7	49	15.6	45.8	
5	3.15	0.22	1560	12.2	5.6	49	14.9	46.0	15.2
6	3.30						16.1	45.2	15.8
6	4.00	0.22	1550	12.0	4.5	50	14.6	46.2	14.0
					4.7				46.2



2900-20

Recovery of CO_2 from Flue Gas

VI Use of the Denver Flocculation
apparatus as an Absorber

Report #1

Summary

The work was carried out on a 3 liter size apparatus. If comparable results can be obtained on a larger scale, then the equipment has a distinct possibility for use as an absorber. Translated to the basis of a standard (dry), 6 cubic foot unit, one inch would produce 2 tons of CO_2 per 24 hours.

The following facts were also uncovered.

a certain minimum speed is necessary — below this the absorption is so poor as to make the machine worthless.

above a fluid rate of 15 ft³

per gal soln per hour, both the
absorptive efficiency and the
sol. cor./gal soln/hr fall off
rapidly

the apparatus develops a
suction of 5" H₂O, no power
would be needed to force
the fluid gas through the liquid

problem - To obtain qualitative and quantitative data on the same apparatus so that the possibility of using this equipment as a absorber may be determined.

Description of Apparatus: The ^(as shown in the diagram) was set up near a source of flue gas (in ch. 14e #2). A 4L aspirator bottle was used to wash out the fly ash and so the gas was drawn in by means of a small displacement pump. It then passed in succession through the gas meter and into the absorber. The rate of flow was controlled by means of a by pass and a screw clamp. Connections were also made for taking of sample

at this point without creating

a back pressure. For the bottles, samples were collected by means of a funnel and a wash bottle. Two turns of "Celrod" were used to keep the solution at the proper temperature.

Procedure: a solution containing approximately 25 gms. $\text{Na}_2\text{CO}_3 / 100$ gms. H_2O was used for the carbonation. The rotor was started and the fuel gas passed in under the following conditions:

Run no.	Fuel gas, psi/hr	Rotor speed	ml color
1, 2, 3	12	780	2900 2900 2900
4	12.0	1340	2800
5, 6	12, 13.5	1550	2700 3300
7	19.7	1560	3200
7(a)	17.0	1560	3200

In all except run 4 (a) the fuel gas was used as it came from the stacks, in the last case, however, air was mixed with the incoming gas so that the % of CO was lower

to 7.97. This was done so as to see what absorption would be obtained using a increased gas rate and two units in cell

The effect of varying rotor speed was tested in runs nos. 1, 2, 3, &

4. a change in the capacity of the

unit was made for runs nos. 6,

7, and 7(a) by putting a plate in

the overflow, the volume of liquid

which could be used was thus

increased from 2700 to 3500 ml

The % of CO in the inlet air

and gas was determined by

was further checked by titration
of samples of the liquid and then

Run #1

Date	Time	Gas Rate, c.f.p.m.	Speed, r.p.m.	Gas Analyser, % CO ₂			Temp, °C	Titration, ml of N H ₂ SO ₄			
				Leak	Exhaling	Inspired		5.0 N	0.1 N	Total	
1	10.25		780				30	21.9	46.1	21.9	46.1
2	10.00	0.15	780	12.0	2.9	8.0	30	21.1	46.4	21.1	46.4
3	11.30	0.20	780	12.0	4.4	8.3	28	20.7	46.4	20.7	46.4
4	12.00	0.205	780	12.0	5.8	9.9	26	20.1	46.4	20.1	46.4
Run #2											
1	11.45		780				35	22.2	46.7		
2	12.15	0.205	780	12.3	8.2	8.3	41	21.4	46.9		
3	12.45	0.15	780	12.0	4.4	8.0	46	20.6	47.1		

Run # 3

Sample No.	Time	Gas Rate, C.F.M.	Speed, R.P.M.	Gas Analysis, % O ₂		Temp, °C	Titration, ml. 0.01N Na ₂ S ₂ O ₄			
				Inlet	Exit by Orsat		Blank	Sample	Total	Ratio
1	10.45							23.5	47.6	
2	11.15	0.20	770	11.9	1.7	7.2	49	22.5	47.4	
3	11.45	0.20	780	12.4	3.6	9.7	49	22.0	47.2	
4	11.50	0.20	780	12.4	2.9	9.4	50	21.6	46.9	
5	2.16	0.20	785	12.4	3.6	11.3	50	21.5	49.0	
6	2.16	0.20	790	12.4	4.8	10.7	50	20.9	49.0	
7	3.30	0.20	790	12.0	5.4	10.0	48	20.5	49.6	
8	3.15	0.12	780	12.2	5.2	10.9	50	20.1	49.5	

Run # 4

Sample No.	Time	Gas Ret'd, C.F.P. (M)	Speed, In. P. M.	Gas. Analysis, % CO ₂		Temp, °C	Total time, min. of N ₂ O		
				Inlet	Exit (Oreant)		S.I.U.F. (M)	M.O.	T.O.F. (M)
1	1.1.0						2.1.2	4.6.4	
2	2.1.0	0.2.0	12.7.0	12.0	3.7	6.2	19.4	4.6.8	
3	2.1.0	0.2.0	12.9.0	12.2	6.2	8.1	18.0	4.6.2	
4	3.0.0	0.2.0	12.9.0	12.2	6.2	7.5	16.5	4.5.6	
5	3.5.0	0.2.0	13.2.0	12.2	6.6	8.4	15.4	4.5.2	
6	3.4.5	0.2.0	13.4.0	11.9	6.5	6.8	15.3	4.2.0	4.5.1
7	4.1.5						4.9	4.6.1	1.6.2
7 (M)	4.1.5	0.2.0	13.4.0	11.3	14.7	6.6	5.0	1.5.4	4.2.3

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Run 25

Sample No.	Time	Gas Rate, c.f.p.m.	Spaced, p.p.m.	Gas Analysis, % CO ₂			Temp, °C	Titration, ml. of N.H. ₄ SO ₄		
				Inlet	Exit (Oxygen)	Corrected Exit		0.01	N.O.	Total
1	10:45						50.1	20.0	45.5	
2	11:15	0.20	152.0	12.1	3.4	4.6	50.1	18.0	45.6	
3	11:45	0.20	182.0	11.8	5.2	6.3	51	16.2	45.5	
4	12:15	0.20	157.0	12.1	5.9	7.3	49	14.2	45.4	
5	12:30	0.22	155.0	12.2	6.6	8.2	53	14.2	45.2	
6	12:45	0.23	150.0	11.9	7.2	5.6	56	15.9	40.8	13.3
7	1:00							15.7	43.0	15.1
7(a)	1:30	0.21	155.0	12.1	5.6	5.2	48	15.1	41.2	13.2

45.6

Runs Nos. 7 and 7^(a)

Time	Temp	Generator	Speed	Gas Analysis		Temp				
				Inlet	Exit (Ore)	Corrected Exit				
1.6	10.15									
2.4	10.45			12.4	11.0					
3.5	11.15			12.1	4.1					
4.4	11.45			12.0	4.5					
5.5	11.45			12.0	3.8					
6.1	11.50			12.1	6.6					
7.1	11.55									
7.4	3.45			11.8	4.4	8.0				
8.4	4.10.00									
8.40	4.10.00			7.9	3.0	5.3				

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M.O. 47.4 11.9 70
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M.O. 47.8 11.5 70
2.7 70 Naitoo

4 - O.C. 21.6 48.3
M.O. 48.3 11.3 70

5 - O.C. 21.1 49.0
M.O. 49.0 11.1 70

6 - O.C. 20.8 49.0
M.O. 49.0 11.1 70

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M.O. 49.6 11.3 70

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Figure 1

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2.00

24.6 gms

Ala CO₂

7 (12.00)

11.3

10.6 11.4 11.4

2.2 3.2 4.4 6.4

7.2 17.6 15.0

0.20 0.20 0.20

13.40

16.2

45.6 7.1

16.1

16.1 15.1 42.3

46.1 16.0 23.8

11.8

4.2 5.0 8.2

14.8

45.2

15.3

0.20

(Run # 4)

1.4 x 0.415 = 0.581 gas CO₂

0.581	gas CO ₂	27.00	total gas CO ₂	0.0345	all CO ₂
1.00		4.5			

CO₂ by gas analysis

6.0	total H ₂	5.2	H ₂ CO ₂	6.0367	all CO ₂
1.00		8.5			

0.0345 x 8.5 = 0.293 H₂ CO₂

0.293 x 1.00 = 4.97% CO₂

6.0, absorbed

$$\frac{2800}{3800} = 0.737 \text{ gal}$$

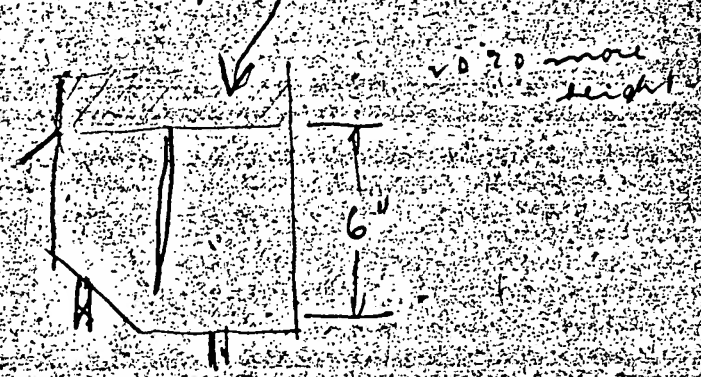
$$\frac{0.033 \times 2 \text{ lb CO}_2}{0.737 \text{ gal air}} = 0.09 \text{ lbs. per gal. air}$$

$$= 155.5 \text{ lbs. CO}_2 / \text{unit / hr}$$

$$= 3770 \text{ lbs. CO}_2 / \text{unit / 24 hr}$$

	11.6	5.2
Coal gas	6.490 cbr	31.6
		45.90

0.2 c f air



5.7	1.8	2.5	2.5
2.9	2.9	7.7	2.7
4.7	1.4	8.9	8.9
0.7	6.0	9.8	9.8
510.0	600.0	800.0	800.0
800.0	00.0	00.0	00.0
4.1	2.2	0.2	0.2
1.5	1.5	2.2	2.2
8.9	1.5	2.4	2.4
1	3	2	2

66
8/17/77

5
6.8
1.0
0.42
0.007
5.2
2.4
6.9
1.8

6
8.5
7.8
0.7
0.49
0.017
5.1
4.8
5.5
6.2

7
7.1

7.4
8.5
7.1
1.4
0.52
0.013
6.6
4.7
4.7
6.6

April 4, 1941.

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Harry Gold

HR:ME

Chemical Department.

all
5/7/7

[illegible]

5.1	6.3	1.7	1.1
2.8	5.1		6.1
2.3			
0.96	0.71		0.1
0.063	0.019		0.1
8.7	6.6		6.6
7.5	5.5		5
3.4	5.2		5
4.6	6.3		4.7

05/9/9

0.3
0.5
0.6
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5

4.7

4.3

7.3

7.7

0.57

0.83

2.2

7.7

6.9

7.7

6

Jerry Gold
June 30, 1938.

RECOVERY OF CO₂ FROM FLUE GAS

IV. USE OF THE TURBO-MIXER AS AN ABSORBER

Forward:

The experimental work recorded here (in Report Nos. 1, 2 and 3) should not be taken as an absolute measure of the value of the mixers because no power readings could be made; this factor may possibly be prohibitive as a very high speed is necessary. However, a high power consumption for the mixers may be somewhat balanced by the fact that no pressure would be required to force the gas through the liquid.

The only bases of evaluation that could be taken then, are (1) the apparent absorptive efficiency as determined by the value of the entrance and exit gases and

(2) the lbs. CO₂ absorbed/gal. of absorber volume -- and even this does not have any meaning.

**USE OF THE TURBO-MIXER AS AN ABSORBER UNLESS
THE INVESTMENT COST OF THE EQUIPMENT PER POUND
OF CO₂ IS KNOWN.**

Report #1

Problem: To try a series of Turbo-Mixers as an absorption unit and to obtain quantitative data so that the value of this apparatus may be determined.

Description of Apparatus:

Three turbo-Mixers were set up in series (as shown in the diagram) in Pyrex Glass Jars of 9 liters capacity each. The drive was provided by a 1/2 HP, 1750 RPM motor and the use of a system of belts and pulleys. A board and two wooden horses were used as a support for the system. The rate of flow of the flue gas was recorded by a gas meter, and the % of CO₂ in the entrance and exit gases was determined by an Orsat Apparatus. The details of the mixers are also given and the passage of the gas and the liquid through them. The red lines show the flow of the gas and the blue the passage of the liquid.

Procedure:

The complete apparatus was set up as shown and each of the glass jars was fitted up to the casing of the impeller with an Na₂ CO₃ solution, containing approximately 25 grams/100 grams H₂O. This is equivalent to 5" in depth, or 45 liters of solution per mixer. Flue gas from the stacks was passed in counter-current to the flow of the liquid and determinations made of

- (1) the % of CO₂ in the entrance and exit gases.
- (2) the rate of gas flow
- (3) the temperature of the solution.

Samples of the solution were also taken and titrated later for carbonate and bicarbonate. The run was continued for 1 1/4 hrs. until a heavy precipitate of NaH CO₃ was obtained.

TIME PM	GAS ANALYSIS % CO ₂		GAS FLOW c.f.p.m.	TEMP. °c.	TITRATION, ml N H ₂ SO ₄				REMARKS
	<u>% CO₂</u>				<u>Solution</u>		<u>Total</u>		
	Ent.	Exit.			O.C.	M.C.	O.C.	M.C.	
3:00	11.6	6.0	1.50	34	23.4	46.8	23.4	46.8	Belts Slipping
3:15	11.6	5.2	1.50	43					
3:30	11.6	4.2	1.50	46					
3:45	11.6	4.2	1.40	49					
4:00	11.6	4.9	1.40	49					Motor Hot
4:15	11.6	5.0	1.70	48	18.4	43.0	14.1	46.9	Control Gas Leak

Material Balance:

Time, Hrs.	Lbs. CO ₂ by Gas Analysis	Lbs. CO ₂ by Titration	% Diff.
16	0.93	0.87	7

Discussion:

1. Data - This first run may really be considered as a "tune up". In spite of the gas leaks encountered and the difficulty in obtaining a uniform drive, a quantitative material balance shows no loss. However, the belts slipped badly and the average exit gas of 4.6 can be improved upon.

2. Results. The results obtained are tabulated below in comparison with those of the rectangular absorber.

FACTOR	TURBO- MIXER	RECTANGULAR ABSORBER
Average % of CO ₂ in Exit Gas	4.6	3.0
Apparent absorptive efficiency, %	60	81 v
Lbs. CO ₂ absorbed per gal. soln.	0.198	0.139
Lbs. CO ₂ absorbed per gal. absorber space	0.104	0.124

No conclusions should be drawn from these figures; they are merely given so that a preliminary idea of the relative values may be obtained.

Basis: 1 hr.

Harry Gold

(June 30, 1938.)

USE OF THE TURBO-MIXER AS AN ABSORBER

Report #2

Problem:

To continue the work using a series of Turbo Mixers as an absorption unit; toward this end it is necessary to set up the apparatus so that more exact information may be obtained.

Description of Apparatus:

The assembly is the same as was used in Report #1, except that an effort was made to cut down the leaks by putting in a new gasket of softer rubber between the jar and the body plate of the mixer.

Procedure:

No change was made from the method used in Report #1, aside from "time outs" of one hr. or more after each 30 minute period of operation to

1. patch leaks
2. take samples of the solution
3. add fresh alkali when necessary.

Considerable slippage was still encountered with the belts, especially after the mixers had run for 20 minutes in each period of operation. More complete data was taken than in Report #1, particularly on the Na_2CO_3 soln. As was mentioned above, when precipitate appeared, the carbonate content of the solution was raised by the replacement of 2 liter portions of the slurry by fresh Na_2CO_3 solution at 1/2 hr. intervals. The gas flow ranged between 1.20 and 1.75 c.f.p.m. but for the most part was kept fairly constant at 1.50 c.f.p.m. - the upper and lower limits were to test the effect of varying rates. The total actual time for the run was 2 hrs.

DATA

Time PM	Gas Analysis		Gas flow c.f.p.m.	Temp, °C.	Titration, ml. N H ₂ SO ₄			Remarks
	Ent.	Exit.			Soln.	H ₂ O.	Total	
3:35	13.0	4.8	1.55	45	24.0	48.0	24.0	48.0
3:40	13.2	4.4	1.55	47				1 1/2 hr.
3:55	14.0	5.2	1.60	49	19.9	47.3	19.9	47.3 operation
5:00	14.0	6.0	1.75	45				
5:20	13.8	5.4	1.45	49				ppt.
5:30	13.2	4.6	1.45	49	16.8	44.7	16.8	47.5 + 8 g. operation
6:10	14.0	4.4	1.50	46				N ₂ CO ₂ soln.
6:25	13.2	4.0	1.40	48				
6:40	13.9	4.4	1.20	49	14.4	39.8	14.4	48.4 1 1/2 hrs. operation
11:00	14.1	5.8	1.75	50	15.7	41.4	15.2	47.6 + 2 g. N ₂ CO ₂ soln.
11:15	14.0	4.8	1.30	50				
11:30	13.8	4.3	1.50	50	11.8	33.9	10.7	47.8 2 hrs. operation

Material Balance -

Time Hrs.	Lbs. CO ₂ by Gas Analysis	Lbs. CO ₂ by Titration	% Difference
1/2	0.48	0.46	2
1/2	0.46	0.39	18
1/2	0.41	0.39	5
1/2	0.48	0.49	2

Discussion:

1. Data - The check on the absorption by titration of the solution shows very little loss of gas by leakage. This, however, is due entirely to the pains taken to patch the bad spots and does not correct the inherent fault of the gasket and the warped body plate.

2. Results - These figures are obtained from the data after 1 1/2 and 2 hrs. of operation; that is, in both cases when precipitate was present as would be in the large scale recovery of CO₂. The results for Report #1 are given and also those for the rectangular absorber.

Factor	Turbo-Mixer		Rectangular Absorber
	Report #1	Report #2	
Average % of CO ₂ in exit gas	4.6	4.4	5.0
Apparent absorptive efficiency, %	60	68	81
Lbs. CO ₂ absorbed per gal. soln.	0.198	0.248	0.139
Lbs. CO ₂ absorbed per gal. absorber space	0.104	0.130	0.124

These show that:

- a. The apparent efficiency is not quite as good as that of the regular process, in fact it is 20% less, but this can be improved upon.
- b. The mixers will absorb approximately 1 1/2 times as much CO₂ per unit volume of absorbing solution as the rectangular unit, but this should be compared with the fact that,
- c. For a series of mixers this figure is only 5% greater on the basis of CO₂ absorbed per unit of absorber vol.

It was also discovered that increasing the rate of flow of gas above 1.50 c.f.p.m. (equivalent to 0.0675 f.p.m.) decreased the absorption considerably, so that the exit gas went up to 5.8% CO₂.

Summary:

The mixers show a definite possibility for use as an absorption unit (this is not considering the limited field of the data obtained); on the basis of CO₂ absorbed per unit vol. of absorber space it compared very favorably with the rectangular unit. The trouble encountered in continuous operation was still not ironed out, tho this only affects the small experimental apparatus.

June 30, 1955.

USE OF THE TURBO-MIXER AS AN ABSORBER

Report #3

Problem:

To continue the runs, using a series of Turbo-Mixers as an absorptive unit, with the purpose of

1. Improving the apparatus so that it would require less attention to leaks and the drive.
2. Determining the amount of CO₂ removed by each mixer.

Description of Apparatus:

An attempt to eliminate the gas leaks was made by putting a heavy layer of litharge and glycerine between the top of the glass jar and the rubber gasket; otherwise the apparatus was the same as in Report #2.

Procedure:

The identical method was used as in the previous work and the absorption was continued until a precipitate appeared. Then 2 liters portions of the slurry were replaced by fresh Na₂ CO₃ solution and determinations made of the absorption in each mixer. The total time of absorption was two hours.

DATA

Time P.M.	Gas Analysis % CO ₂		Gas Flow C.F.P.M.	Temp. OC.	Titration, ml. ^N H ₂ SO ₄				Remarks
	Ent.	Exit			Soln.		Total		
					O.O.	M.O.	O.O.	M.O.	
2:40	12.8	8.2	1.55	40	24.5	49.0	24.5	49.0	No speed
2:50	12.9	2.4	1.50	45					High speed
3:00	14.0	2.0	1.70	45					Leak
3:10	14.0	1.0	1.60	45	21.4	49.3	21.4	49.3	Leak, 3 hr.
3:45	14.2	5.0	1.40	41					Low speed
4:00	14.0	3.5	1.70	48	19.1	49.2	19.1	49.2	3/4 hr.
7:00	13.6	2.2	1.20	45					
7:15	13.7	2.4	1.20	45	17.6	44.5	17.2	49.4	1 hr.
8:30	13.7	10.0	1.55	42					1st mixer + 24 Na ₂ CO ₃
8:40	13.9	5.2	1.55	43					2d mixer
9:00	13.7	2.0	1.55	45					3d mixer
9:20	13.6	9.7	1.50	48					1st mixer + 24 Na ₂ CO ₃
9:40	13.6	6.0	1.45	48					2d mixer
9:50	13.6	2.2	1.45	48					3d mixer 2 hrs.

Material Balance -

Time Hrs.	Lbs. CO2 by Gas Analysis	Lbs. CO2 by Titration	% Diff.
1/2	0.45	0.40	?
1/4	0.28	0.29	3
1/4	0.24	0.24	-

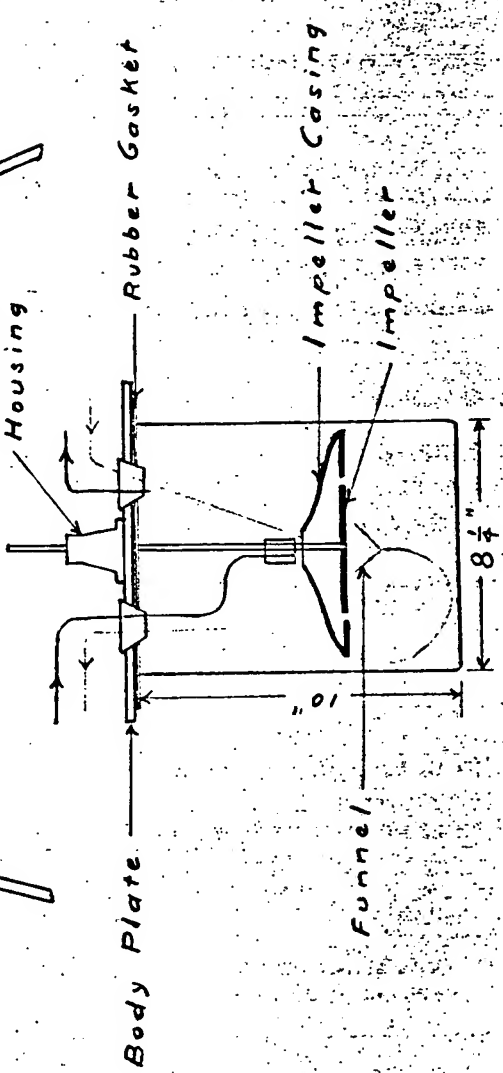
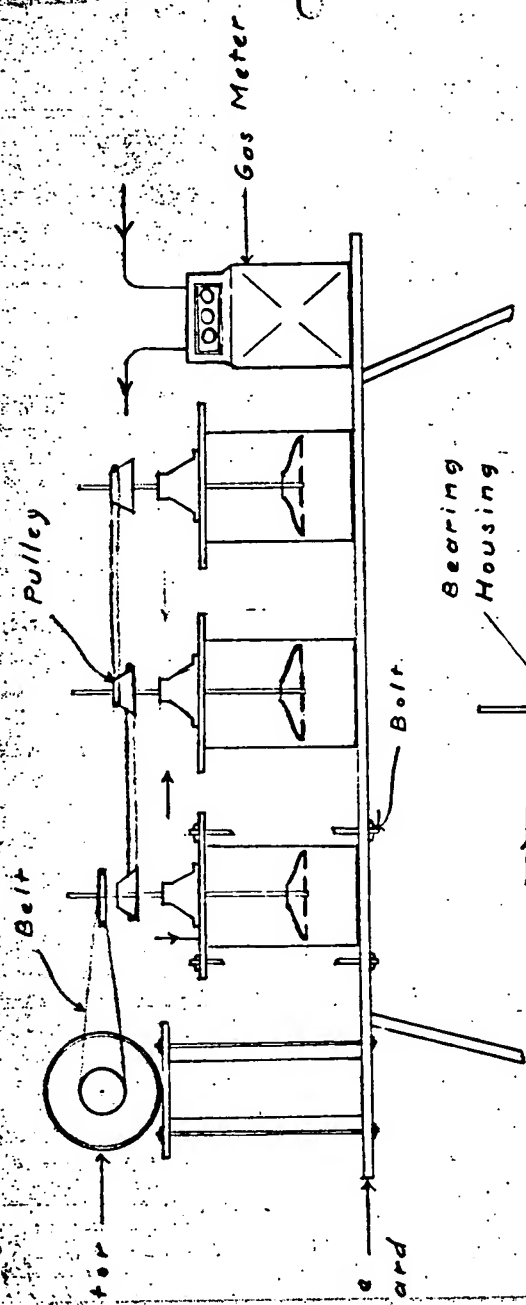
Discussion:

1. Data - The material balance on the absorption shows very little loss but the seal of litharge and glycerine blew out continually and time had to be taken to patch it. The difficulty was finally traced to the following sources:

- The slight warping of the cast aluminum body plate.
- The insufficient number of bolts (5) to pull down evenly on the body plate.
- The need for a very soft rubber gasket of either pure or sponge rubber.
- Especially, the lack of a by-pass for relieving the pressure when a sample of the exit gas is taken.

It was also decided that the drive could only be made more uniform by mounting the entire apparatus on a sturdier and more workmanlike base than heretofore used - makeshift devices had failed to correct the essential faults and the only reason the absorption was improved over the first two runs was due to the greater experience in operating the unit. The greatest troubles here are

- The horses with the board across them form a support which is entirely too light and which has no real rigidity.
- The shafts of the mixers were only fixed at one place - the bearing housing on the body plate - and as a result the pulleys on these shafts could be drawn together or apart almost as desired. This gave a very uneven tension on the belts with the consequent variability in the speed of the impellers.



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We shall now consider the stability of the individual important vitamins of commerce:

Vitamin A—Vitamin A is slowly destroyed by exposure to the air and light. Even in fish oil, which is our most important source of vitamin A, this destruction is material. To prevent, or at least reduce it, some of the commercial producers of fish-oil concentrate now store the oil in barrels in which they introduce carbon dioxide gas in order to exclude air as far as possible. Strangely enough, carbon dioxide is quite soluble in these oils.

Vitamin A is also destroyed by the enzymes lipase and lipoxidase (which are found in the livers and split oils and fats to form free fatty acids). Therefore, some fish oils which are produced by autolysis (i.e., by letting the fish liver decompose in the sun until the oil cells are so tender that the oil can be pressed out—resulting in exposure to this enzyme action) frequently are completely void of the vitamin. These active enzymes can also be produced by some micro-organisms. Some seeds contain enzymes which are able to destroy vitamin A (carotene) when fed in the same ration. These enzymes may be present in dried milk, fish and meat products. In some cases these enzymes are active in the dry state. Heating will in many cases destroy or inhibit such enzymes. Many metals, such as iron, copper and zinc, have a pro-oxidative action and should therefore not be allowed in contact with vitamin-A oil.¹

VITAMINS ON THE DINING TABLE. Increasing interest in supplementing the diet with a daily ration of the B-complex vitamins has been stimulated by the latest discoveries in the vitamin field. Among the many forms and combinations in which this can be taken is B-Nutron Syrup, a high-potency source of the entire complex, obtained from rice bran concentrates and supplemented with synthetic thiamin (B₁), riboflavin (B₂), and nicotinic acid, plus iron in the form of the gluconate (non-toxic). Produced by the Nion Corporation, Los Angeles, it comes in 4-, 8-, and 16-oz. bottles, and a 2-oz. dropper bottle with carton (Angelus Paper Box Co.). Package design and container by Owens-Illinois.

Wide-mouth glass containers are approved by pharmaceutical houses packing vitamin capsules, because capsules in glass keep dry and firm and the wide mouth is superior from a packaging standpoint. These high-potency Vitamin A B G D Concentrate Capsules are produced by Frederick Stearns & Co. of Detroit (one of the earliest concentrate-tablet makers). Round Owens-Illinois jars, holding 25, 100, and 250 capsules,

All light of wave lengths shorter than 5000 A. U. should be eliminated from vitamin products. Some red cellophanes are very efficient filters for the removal of such undesirable light.

Fortunately, there are available many preserving agents which frequently are quite effective; some of them are able to postpone oxidation for several years. Partial hydrogenation² has been tried and is said to be quite effective. The addition of anti-oxidants is the most common procedure. Such agents may be extracted from various seeds. Carrot and soy bean seeds are particularly good.³ Oat flour and soy bean flour are frequently used and have been the subject of many patents. Wheat-germ oil, which is our best commercial source of vitamin E, is also commonly used. Some resins from plants are effective under certain conditions and have been reported to be satisfactory in some manufactured products. Among the chemical anti-oxidants hydroquinone, catechol, pyrogallol and hydroxyhydroquinone are used to some extent.⁴

In considering any anti-oxidant it will be wise to check the toxicity of the finished product. From a chemical aspect an anti-oxidant is apparently just an agent which is oxidized easier than the vitamin, and the anti-oxidant consequently absorbs the oxygen which comes in contact with the product before the oxygen is able to affect the vitamin. The action therefore provides only a temporary postponement of the vitamin destruction—this postponement, however, may be for a period of several years.

Complete exclusion of air, whenever possible, is preferable. Medical tablets have been made, consisting of fish-oil concentrate absorbed by an inert powder and subsequently pressed into tablets and coated with several layers of sugar. Such tablets have been found satisfactory after several years of storage under ordinary conditions.

These oxidation processes are greatly accelerated by temperature. As a result, it is always advisable to store
(Continued on page 307)



How to reduce the instability of vitamin content

(Second article of a series on techniques of vitamin fortification)

By HALFDAN HEBØ, D.Sc., F.R.M.S., New York*

The manufacturer who has considered the question, "Shall we add vitamins to our products?" (subject of the first article in this series) is confronted immediately with a further consideration. This is the matter of vitamin-content retention in the products to be fortified. Dr. Hebo now takes up the use of special processing techniques and the addition of preserving agents to accomplish satisfactory retention.—The Editor.

One of the most difficult phases of food processing is the problem of proper retention of vitamins, whether they be naturally present or added. Vitamin D is the only one which is reasonably stable during ordinary processing and long-period storage conditions. Fortunately, a study of vitamin chemistry provides us with some inside knowledge of conditions which destroy the various vitamins. By avoiding certain conditions in processing, such as high temperatures and oxygen, we are frequently able to prevent material destruction of these vital components.

Vitamins have only one thing in common: their names. Now that the tendency is to call them all by their chemical names, rather than by their letters, it is safe to state that they have really nothing in common. They are not in every case necessary for human nutrition. For example, vitamins B₁, B₂, B₆ are not required by man; and apparently vitamins L₁ or L₂, which support lactation, may be dispensed with even during lactation if certain other food factors are present. Other food factors which are definitely essential for sustaining human life are not known as "Vitamins." Calcium, phosphate, iron, iodine, and amino acids are but a few of several dozen chemicals which we must have in our daily diet to live.

While most of these other essential food factors have fairly simple molecules, some of the vitamins have large intricate chemical formulae. For instance, the vitamin D (calciferol) molecule consists of 70 atoms; vitamin A has 51 atoms; B₁, 38; and C, 20 atoms.

It is quite reasonable to suspect that some of these chemicals may react chemically with each other and form new, inactive compounds. There has even been some talk of "Anti-vitamins"—if we were to accept this term, oxygen

ought to rank first among these "fifth-columnists" of biology, because all of our known vitamins succumb to its merciless attack. Some enzymes are likewise enemies of the frail vitamins and a number of metals and metallic salts destroy or counteract the vitamins. Ultra-violet rays, which are used in creating the D vitamins, destroy all the vitamins including those which are created by it, if exposure to the rays is prolonged sufficiently.

A typical example of vitamin conservation and destruction in various methods of food preservation has recently been reported by Farrel and Fellers.¹¹ They preserved beans by canning, quick-freezing and dehydration. The Bountiful variety of snap beans were quick-frozen and were blanched (scalded) before freezing. The following table indicates the vitamin content before and after processing. After one year of storage the vitamin losses were reported to be negligible.

	Fresh raw beans	Canned	Quick- frozen	Dehy- drated
Vitamin B ₁ (International units)	16	14	19	39
Vitamin B ₂ * (Bourquin-Sherman units)	33	32	39	40
Vitamin C (International units)	400	64	218	18

The blanching process as a preliminary treatment to canning or freezing appears to be of utmost importance in conserving vitamins B₁ and C because it inhibits or destroys some of the destructive enzymes which frequently are present in vegetables, fruits and oils. In this experiment vitamins B₁ and B₂ were tested biologically on rats. The apparent increase of these two vitamins in quick-freezing and dehydrating may be credited to the enzyme-inhibiting treatment (blanching). It appears that these enzymes, if not inhibited or destroyed, are active and may destroy the vitamins after the beans have been consumed by the test animal.

After canning and processing of foods, it is frequently observed that the water-soluble vitamins are present in the liquid rather than solid part of the product. In such cases the consumer should be advised to use the entire content of the package.

A concentrated mixture of all the vitamins is surprisingly stable if no water is present. Such a mixture, consisting of shark-liver oil containing in excess of 100,000 units of vitamin A per gram, mixed with crystalline vitamin B₁, B₂ and C, with vitamin D concentrate added, is used for capsulating and has been found satisfactory. The deterioration under normal conditions is frequently

*Dr. Hebo received a degree in Chemical Engineering from the Royal Polytechnical Institute, University of Copenhagen; and is a Fellow of the Royal Nordic Society at Copenhagen, oldest scientific society in the world. In the last ten years he has specialized in the chemistry of ultra-violet irradiation, as applied to vitamin-content of food and dairy products and in connection with the measurement of vitamin-content by spectroscopy. In 1934, he introduced the Titus Lamp for treatment of erysipelas. Dr. Hebo has been retained in a consultative capacity by leading American food and pharmaceutical concerns.

*A Bourquin-Sherman unit of vitamin B₂ is the amount of the vitamin necessary to produce a specified amount of growth in rats. It cannot be translated directly into weight units and various investigators have reported it equal to from 2 to 7 micrograms.

How to reduce instability of Vitamin content (Continued from page 286)

vitamin-A concentrates and products at as low temperatures as possible and practical.

Vitamin B₁—Vitamin B₁ is more stable to oxygen than vitamin A; but is easily destroyed by mild reduction agents, such as sulphites and tannins, by permanganates, tartrates and manganese oxide; and by hot alkalis. The vitamin in an acid medium is fairly heat-stable and will stand ordinary processing with a minimum loss. For instance, tomato juice heated to 100° C. for four hours with a pH adjusted to 4.28, lost only 20 per cent of its vitamin B₁ content, but with an adjusted pH of 5.2 the loss was 30 per cent and at a pH of 7.9 the loss was 70 per cent.⁵

A weak solution of the vitamin in plain water is not stable and some of the commercial products thus sold, particularly those sold for stimulating plants, have in many cases been found to be deficient in B₁. It appears that the stability of the vitamin naturally found in common foods is greater than in pure form; this has been attributed to a chemical combination it forms with pyrophosphoric acid. In general, it can be stated, it is not necessary to add a preservative agent to foods containing or enriched with vitamin B₁. Even by baking only 5-9 per cent is lost in potency, although in toasting whole-wheat bread as high as 17 per cent sometimes is lost. The best way to preserve the vitamin is to lower the pH concentration of the product whenever possible.

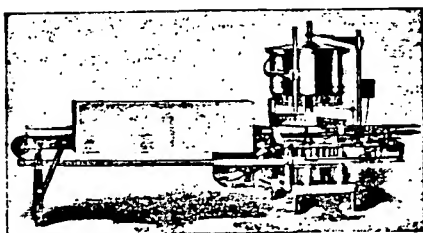
Other B Vitamins—Riboflavin or B₂ is, like vitamin B₁, fairly stable but subject to destruction by reduction. It is stable at higher temperatures and decomposes first at 274° C; but it is readily destroyed in alkali solution, particularly if exposed to strong light, visible as well as ultra-violet. In food processing no noteworthy loss of the vitamin is observed. Due to its relatively high cost thus far, it is infrequently added to food products.

Nicotinic acid, which recently entered the vitamin kingdom, is one of the cheapest vitamins, and is already being added commercially to flour. It is even stable above 230° C, which is its boiling point, so in this respect no precautions are apparently needed.

Vitamin C—Vitamin C is one of the frailest of the vitamins: oxidation, alkali, metals, heat, and ultra-violet light are among its many enemies. The best way of preserving the vitamins is to exclude as many of these destructive elements as possible. At low temperatures in an acid medium, oxidation is insignificant, but at prolonged heating the destruction is rapid. Frozen orange juice loses little of its vitamin-C content during a year of storage, and flash pasteurization in vacuum does not affect the vitamin content materially. Metals, particularly copper and iron, should be carefully excluded from contact with foods containing Vitamin C. A little speck of rust quickly oxidizes the vitamin. Riboflavin in aqueous solution will also inactivate it, particularly if exposed to light. The dry crystal is remarkably stable and will stand exposure to air for years without being destroyed by oxidation.

Ordinary processing destroys about 50-85 per cent of the vitamin C content of foods, but it has been reported that the addition of common salt (sodium chloride) to vegetables in some cases acts as a preservative of the vita-

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min, perhaps because this addition makes oxygen less soluble in the water in which the vegetables are cooked. Many of the enzymes ordinarily found in fruits and vegetables are supposed to be responsible for a material part of the destruction; such losses can be reduced if the foods are placed in boiling water instead of beginning the cooking in cold water.⁶ Processing in vacuum also will reduce the loss considerably. It has been reported that it is possible to can tomato juice without loss of vitamin C at all.⁷

When fruits and vegetables are crushed, enzymes are liberated. This leads to substantial vitamin destruction. In some cases this can be prevented by inactivation of these enzymes by quick-heating before crushing. Processing at low-pH concentration preserves the vitamin-C potency and the canned citrus juices may contain 70-90 per cent of their original vitamin-C content.

Vitamin D.—Chemically, vitamins D₂ and D₃ are so much alike that here they will be dealt with jointly as just "vitamin D." This D can be regarded as one of the most powerful of the vitamins: one ounce of it will cure 40,000,000 rickety rats. It is also one of the most stable vitamins. Under ordinary conditions it will withstand oxidation, heat, and ordinary light—although short ultra-violet rays will destroy its antirachitic power and will under certain conditions form a toxic compound: toxisterol. The vitamin is fat-soluble and it is reported that it is more active if it is dissolved in a non-saturated fat, such as a fish oil.⁸

It might be mentioned in passing that a few years ago it was reported that cereals were rachitogenic (i.e., ricket-creating) and this was blamed on a substance known as phytin. This factor was also called "toxamin" by the American Medical Association's Council on Foods (1937).⁹ Others have reported that beryllium¹⁰ and its salts were rachitogenic. These are the two major factors which affect the calcium-phosphate metabolism despite the presence of a normal supply of vitamin D.

The food-packing industry should not be concerned about these rachitogenic factors at the present time. Beryllium is infrequently found in foods. The phytin problem has not been defined and authorities disagree as to its importance. But it should be pointed out that vitamin D is ineffective *unless the body is adequately supplied with calcium and phosphate salts in a form in which they may be assimilated.* It is therefore advisable to add these salts to food products which have been enriched with vitamin D, when it is practicable and possible to do so. The human body requires a minimum of about one and a half grams of calcium and one gram of phosphorus daily.

This vitamin is destroyed after prolonged heating at 200° C, even if air is excluded.

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- ¹¹Kenneth T. Farrell & Carl Fellers: 101st Meeting Amer-

65-4307
EX 120

c. r. Reaming

1. John family report.

2. $\text{Colum} \left\{ \begin{array}{l} \text{Nucleus} \\ \text{Nucleus} \end{array} \right. + \text{Vsg Nucleus}$

10000

³⁻⁷
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of the past the... down eyes for

a like quest -
C.S. in some who showed down eyes from
... due to the

3. This may very likely be due to the action of the alkali on the water parking and considerable oil was dissolved into the solution.

Proceeded: and 8th steam low to 10 in steam

<u>CO₂</u>		<u>Mass</u>
105° - 1 hr =	40 gcs.	12 lbs
110° = 1 hr =	60 "	6 "

(125 cc)

60 gcs compression

6.7 gcs Na₂CO₃ in sol.

10.0 NaHCO₃

76.7 gcs =

6.7 gcs Na₂CO₃

71 gcs NaHCO₃ = 18.5 gcs CO₂

120 cc = 60 min = 3.5 gcs CO₂

20% CO₂

2% - sol

87% more to decompose

6.7 gcs Na₂CO₃

8.5

15.2 gcs Na₂CO₃

It is not necessary to ~~state~~ ^{obtain} a heat balance which
 then determines the form
 of the apparatus & the manner
 of decomposition.

1/1/50

with on (C.O. or heavier) 0 # ✓ 9/29/57
Data for - 1 ton plant

The information we have obtained so far is more qualitative than quantitative due to ~~various~~ ^{inherent of unavoidably} variable factors in the expts. The following ^{points must} ~~data should~~ be noted

1. ~~one~~ very careful on carbonation run running

- a. ~~vs~~ ^{vs} Al_2O_3 / 100 gms H_2O
- b. ~~at flow of~~ 900 c.f.t. p.d. ~~of~~
- c. a carbonation temp of $\approx 118^\circ\text{C}$.

From this point to which it is feasible to carry the carbonation should be very carefully ~~observed~~ ^{followed} as this point ~~determines~~ ^{fixes both} the manner of carbonation and the composition of the dissociation mixture. This point is more fully discussed in note # 1.

2. Decomposition data at 110° , 105° , and 110°C using v r and

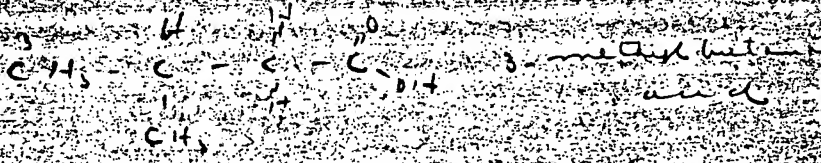
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General Formula

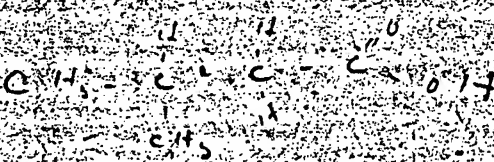


nomenclature

1. Acetic

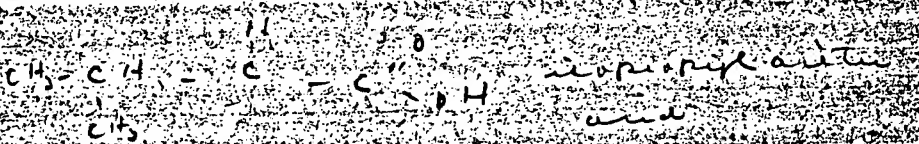


carboxylic acid



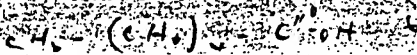
2-methyl-3-oxopentanoic acid

3. Amino acid derivative



formic
acetic
propionic
butyric
valeric
caproic

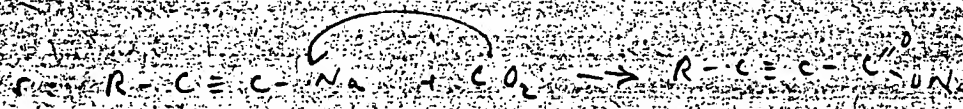
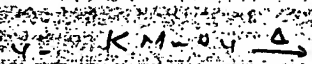
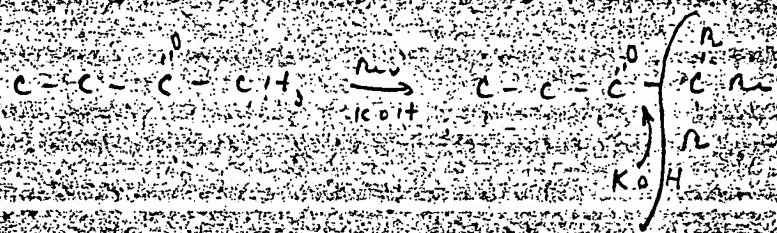
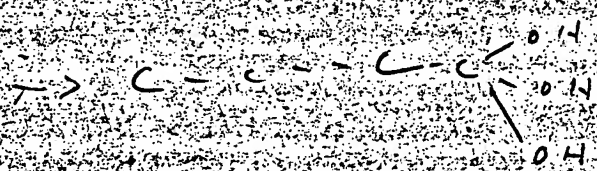
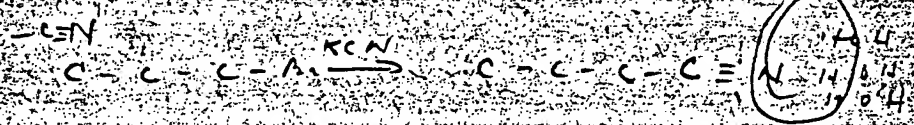
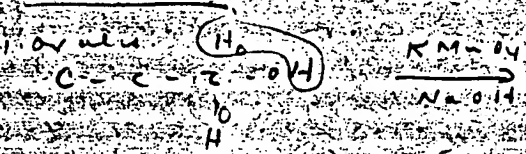
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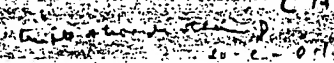
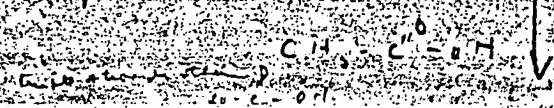
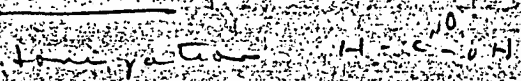


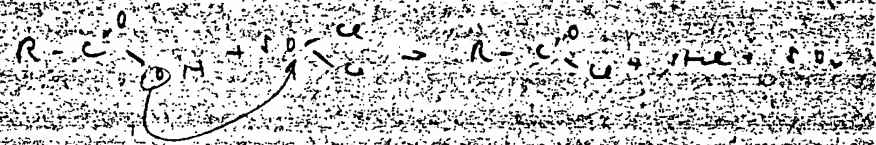
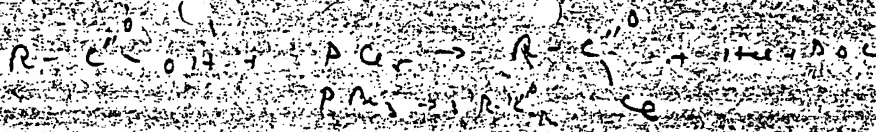
γ-methyl butyric acid

Synthesis

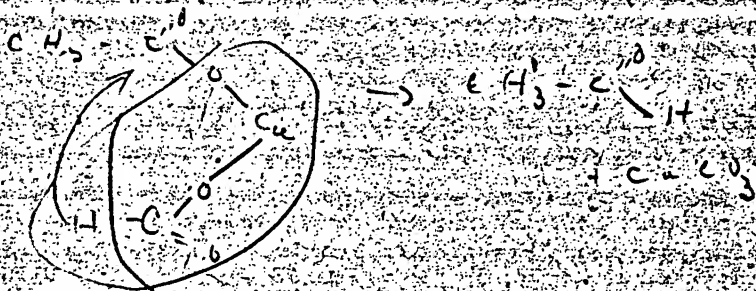
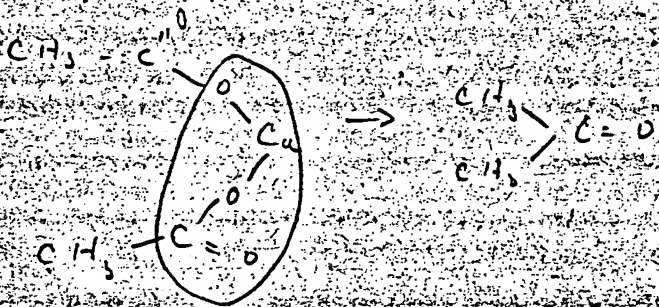
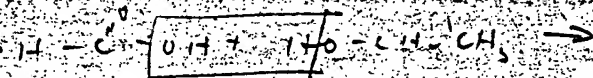


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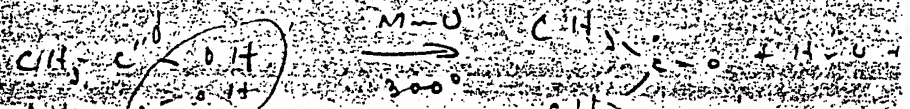




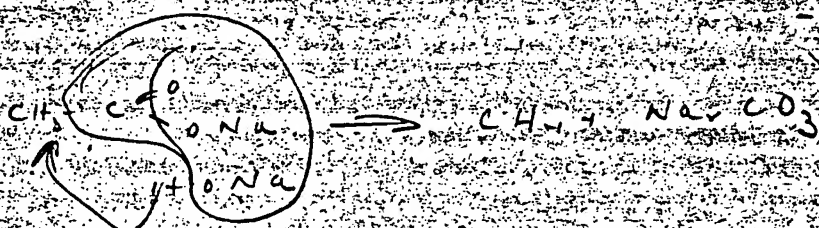
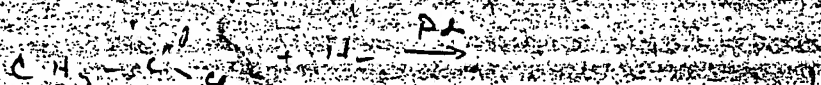
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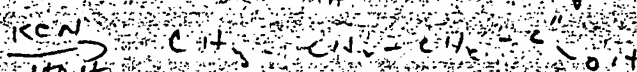
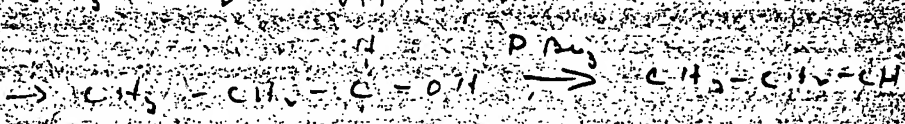
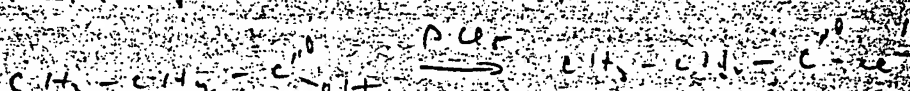


6. cannot oxidize, reduce or hydrolyze
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Special

1. Esterification



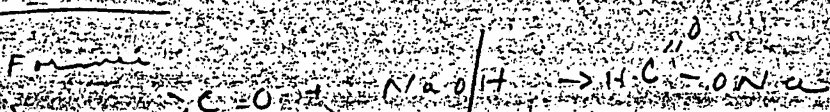
2. Separation of acids

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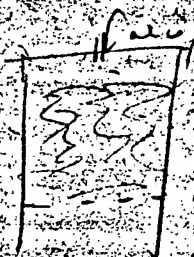
(e) Fractionate

Individual acids

Formic



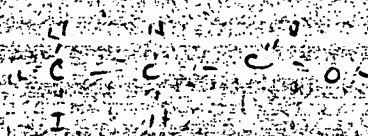
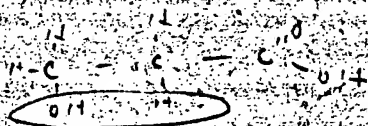
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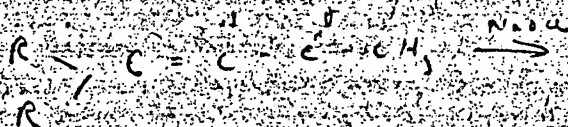
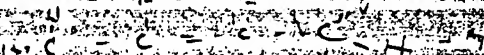
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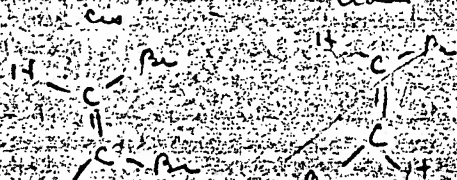
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1) acid cond



monomer (cat)



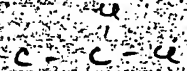
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alkyl radicals, telomer

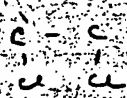
$C-C-C-C$ - n-propyl chloride

Poly-



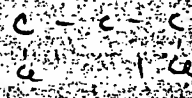
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ethylene
chloride

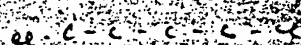


vicinal

ethylene
chloride



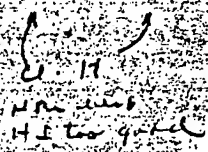
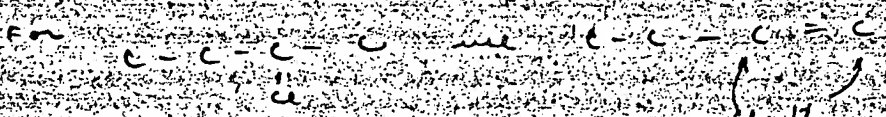
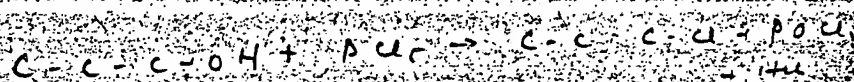
separated
disposition



α, β -disubstituted

monochloride (rare)

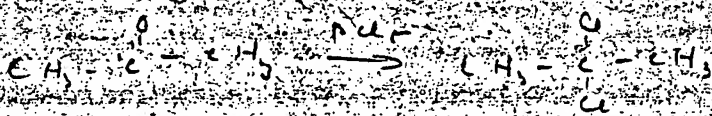
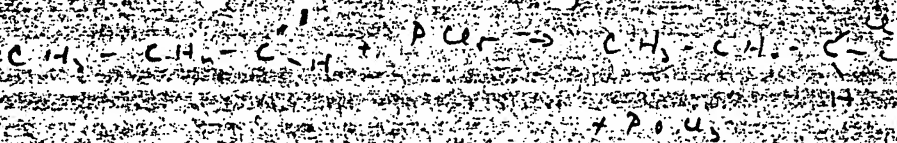
alcohol + PCl_5 [focul, PCl_5 , telomer]



non auto
H to q-ful



CH_3-C-Cl from con. alc, gas on solid

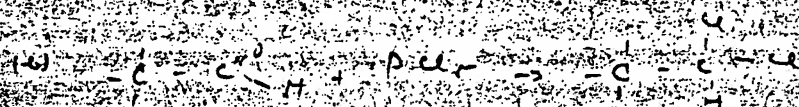
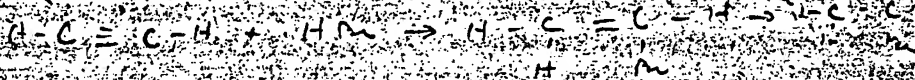


can produce an ester

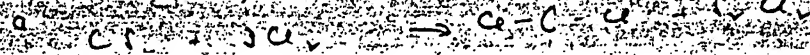
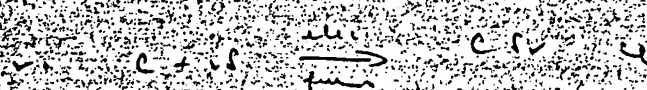
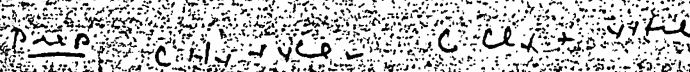
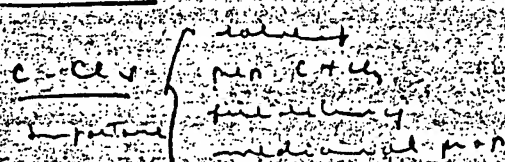
alcohol

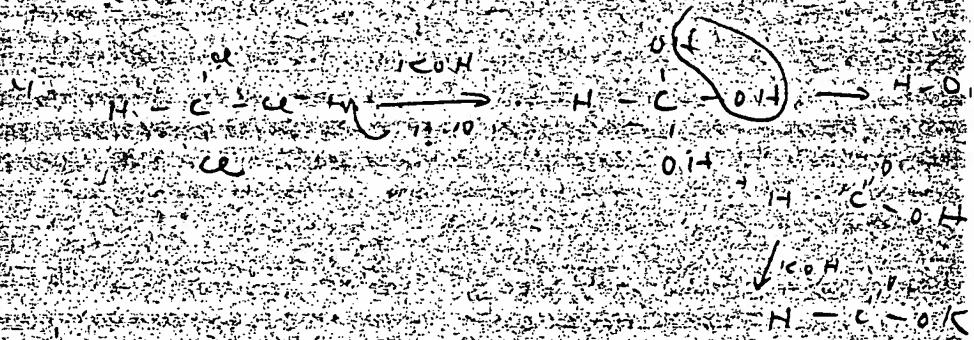
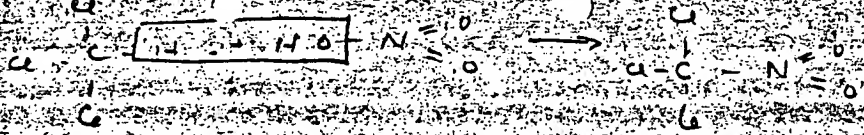


then

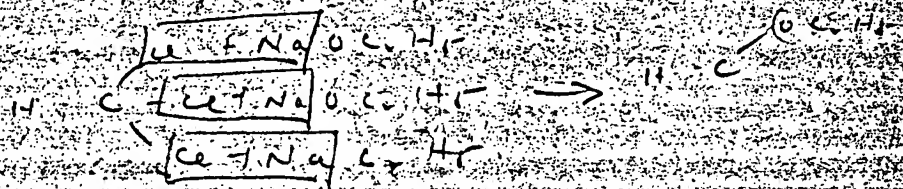


Special Cases

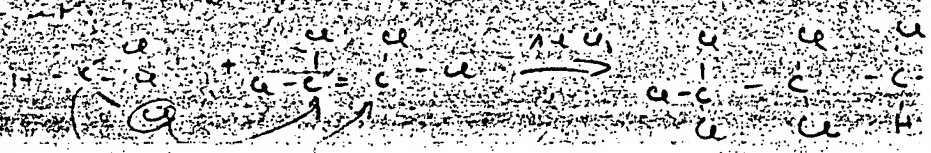
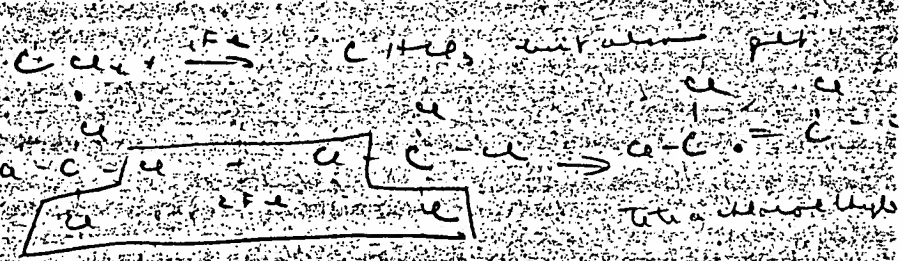




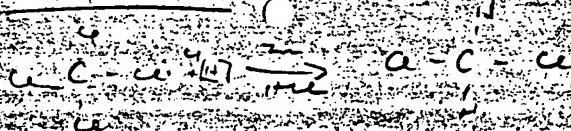
ethyl ortho formate



addition reactions



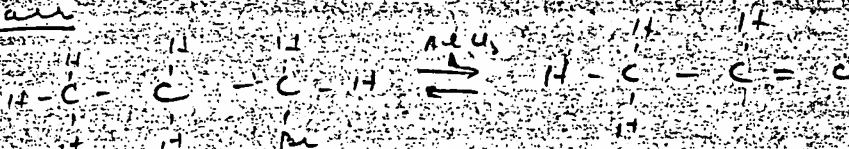
Reduction



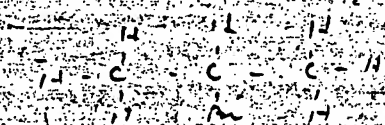
methylethyl chloride

but, C_4H_{10} much more comp.

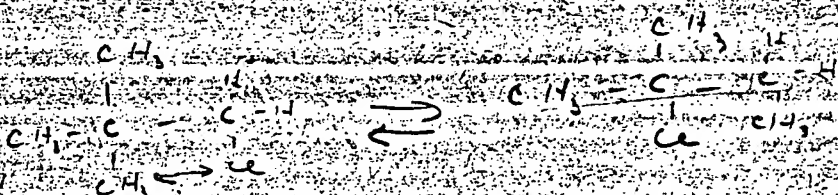
Rearr



aceto rule



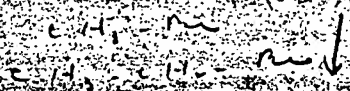
also

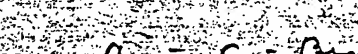
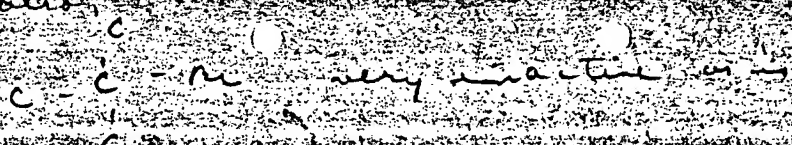


2 methyl 2 chloropropane

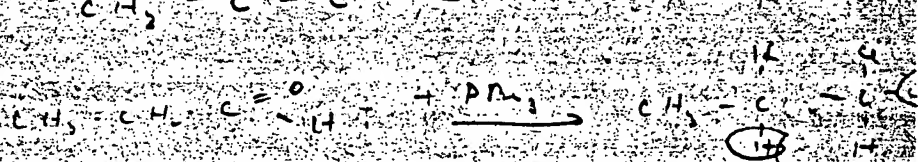
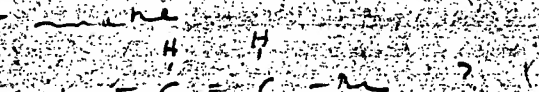
Reactivity

rate depends on length of R

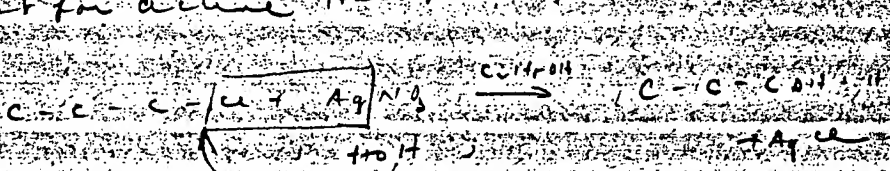




How make



Test for active Halogen



Cyanides and nitriles

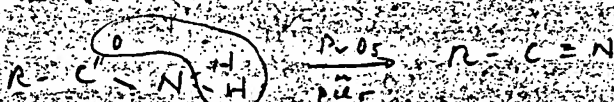
Structure

$H-C \equiv N$ hydrogen cyanide / formonitrile

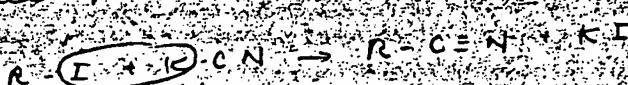
$CH_3-C \equiv N$ methyl cyanide / acetonitrile

Preparation

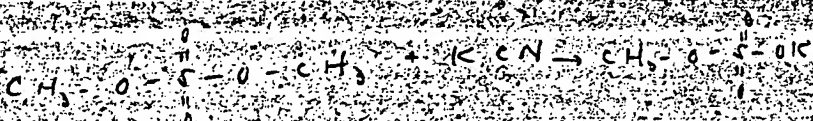
Dehydration of an amide



alkyl isocyanide $R-CN$



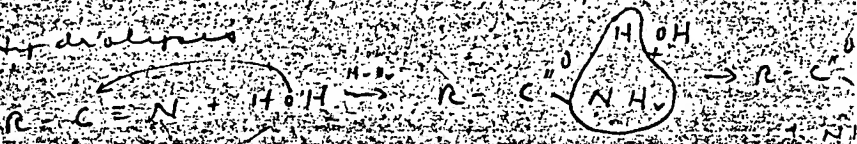
3. For ethyl and methyl cyanides the cheaper alkyl sulfates are used.



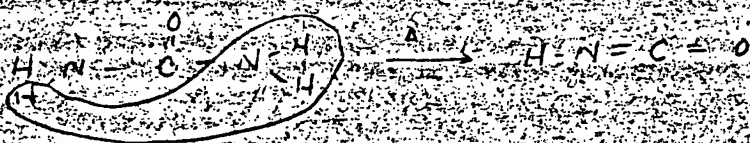
alkyl groups with alkyl groups react

Reactions

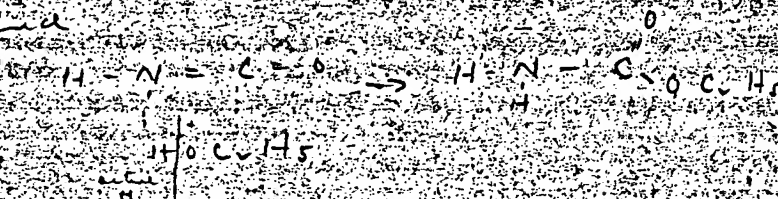
Hydrolysis



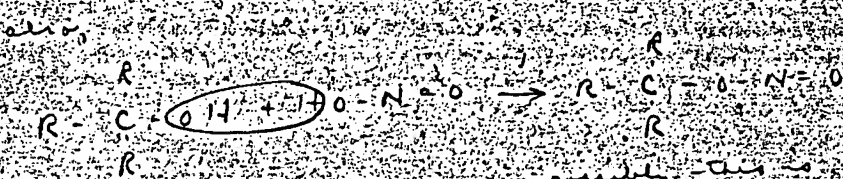
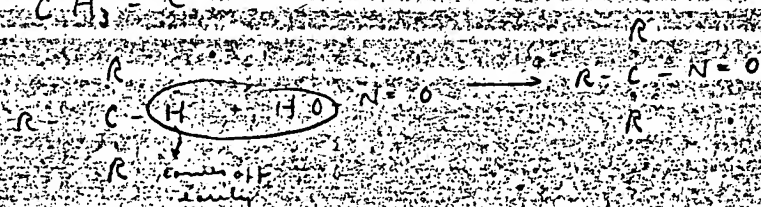
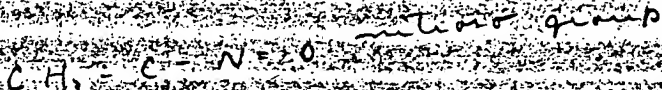
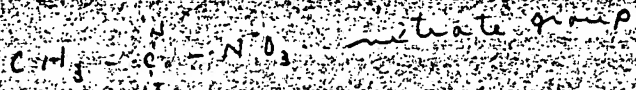
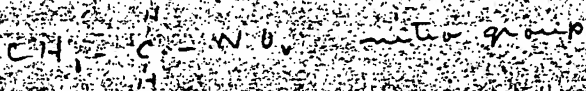
d. prop-1-ene (another way)



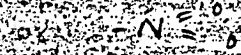
and



2. nitro compounds

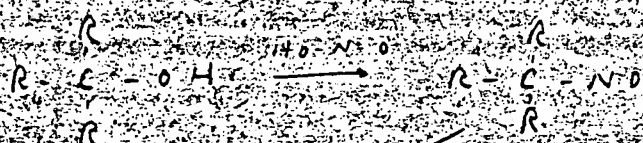


usually as



Reaction

a. all 1°, 2°, 3° alcohol

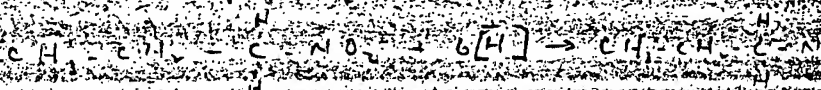


nitration

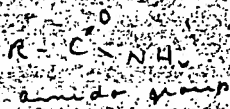
+ KNO_3 , then add H_2SO_4 drop by drop

→ { oxidized → primary alcohol
blue to green → sec alcohol
no color → tertiary alcohol

b. Reduction

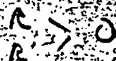


in presence of (acid)



Carboxylic Acids

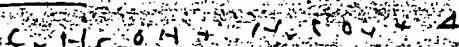
General Formula



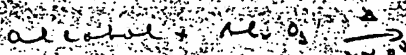
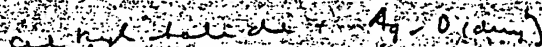
Nomenclature

named from alkyl group
oxidation

Synthesis

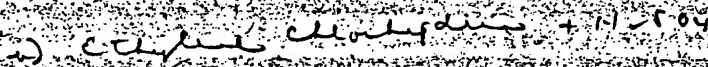


Williamson



epoxide

2) per ethylene oxide reaction with
alcohol + glycol



3) decarboxylation

Chem Prop

hydrolysis

1) reacting with H_2

2) PCR

3) Oxidation with (KMnO₄)

acid + alk

General Formula



Nomenclature


named from alkyl group

1. α -hydroxy aldehyde $[R \cdot C(OH) \cdot C(=O) \cdot R']$ ketone with α -hydroxy
 2. Cu (alkylcuprate) ketone with α -hydroxy
 3. alkyl cyanide ketone with α -hydroxy
 4. $H-C \equiv C-H \xrightarrow{H_2SO_4} CH_3-C(=O)-H$

Reaction

1. no reaction
2. $NaCl$
3. oxidation
4. hydrolysis
5. reduction Na/H

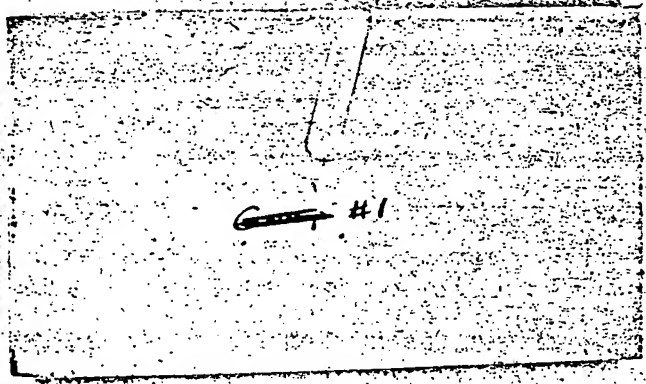
addition Reaction

1. HCN
2. $NaHCO_3$
3. H_2NH_2
4. $H-N(OH)-H$
5. H_2N-NH- 
6. $NH_2-C(=O)-N-NH_2$
7. $H_2O-N=O$
8. $C_2H_5-OH \rightarrow$ acetone
9. PCl_5
10. ~~no reaction~~

65-4307

6/13

65-4307-200



A. BRUCHMAN & ASSOCIATES)

No. 1 of

Date: 10-22-44

JOB:

By: H.G.

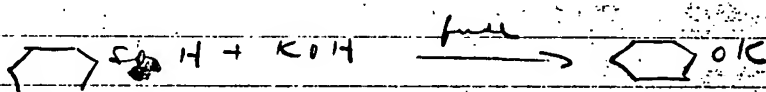
SUBJECT:

Vanillin

I oxidation of to C^1H

Reaction temp about 120-140 will
 vol II C^1H under carefully
 controlled conditions, but
 → no references.

Reaction, 170 vol II



A.C.A. 1930, 980 Thorne I 664

{ J. Chem Ind., Russia, 1929, 6, 1439-1440
 (S.S. Shenderovich & S.S. Livshits) }

The optimal conc of acid is 60%,
 rise of temp favors the formation of tars,
 while below 350 the reaction is slower
 & more benzoic acid is formed. An
 excess of pyrolysis may cause the
 formation of tars during distillation
 with steam. Prolongation of
 the reaction tends to increase the
 relative quantities of benzoic and
 a tar.

A. BROTHMAN & ASSOCIATES

No. 2 of

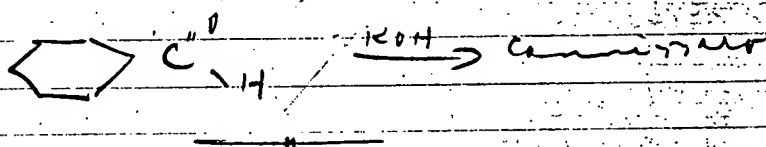
Date: 1.8.44.47

By: H.B.

JOB:

SUBJECT:

Vanillin



methylation of alcohol.

Satterman - Whitland p. 244

1. Dissolve 0.2 mole alcohol 199. in 100 cc of 2 N. NaOH in a narrow necked 9-5 bottle & add about 1/2 of the 6.9 g. of $(\text{CH}_3)_2\text{SO}_4$ to be used (add in 1 lot).

2. Stir \rightarrow cool. of heat.

3. add 2nd 1/2 of $(\text{CH}_3)_2\text{SO}_4$ after 1 min & stir.

4. after "short time" add last of $(\text{CH}_3)_2\text{SO}_4$.

5. Test to see if ag when in which anionic plants is no longer acid.

6. Transfer to disto flask - use 50 cc of NaOH soln as wash.

7. 5 or more wash with for 1 hr. to complete reaction & to destroy

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

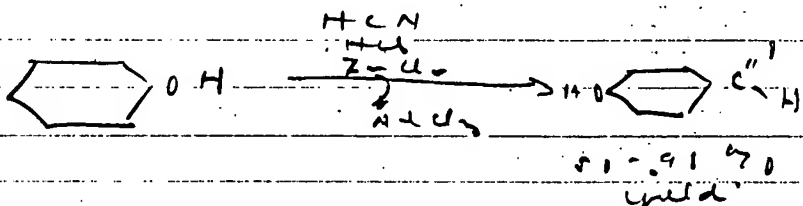
Vanillin

Date: 11-22-47

By: H. G.

the mechanism of Homocoupling
reactions

p. 123.



Satterman Ber 31 1765 (1947)

32 278, 284 (1949)

ann 357 313 (1907)

organocyanine j. chem soc 119, 177
(1941)

Karrer Helv. Chim. acta 2, 89 (1911)

adamson Ber 31 45, 2373 (1943)

adamson & mat " 41, 1512 (1941)

Richter V.H. II p. 301

1. all phenyl aldehydes show the same reactions of the aldehyde group as the benzaldehydes.
2. oxidizing agents convert them with difficulty into phenyl-carboxylic acids.

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: _____

Vanillin

Date: 10-22-47

By: H.B.

1. this is most easily accomplished by
 1. fuming with caustic alkalis
 2. a oxidation with dilute alk. H_2O_2
 the o- & p- phenol aldehydes split off
 the aldehyde group & readily pass
 into pyro-catechins & hydroquinone
 (C. 1911. I. 634)
 4. & they dissolve in alcohols, forming salts, e.g.
 $NaO \langle \text{benzene ring} \rangle C \equiv H$; the alkyl iodides
 convert the latter to alkyl ethers

Battermann Synthesis of Hydroxyaldehydes

J. a. c. s. 41, 2573 (1923)

R. Adams & J. Battermann

1. a. & L. state that big disadvantage
 of method is use of anhyd. HCN .
2. Kauer gets around this by using
 $AsCN$ but yields are poor.
3. a. & L. reverse $Zn(CN)_2$ & use this
 method - $Zn(CN)_2$.

a. says that $NaCN$ (10% soln. 90 pure)
 in 25%, more than enough - out of 140

A. BROTHMAN & ASSOCIATES

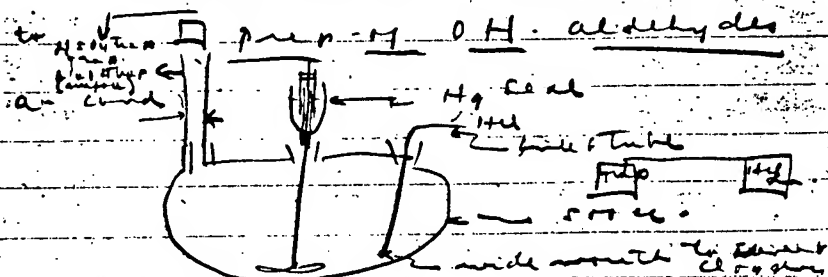
JOB: _____

SUBJECT: Vanillin

Date: 11-22-47

By: H. G.

- b. add $MgCl_2$ soln. until no more ppt of $Mg(OH)_2$ & $MgCO_3$ forms. (this requires an amt. of $MgCl_2$ sufficient to ppt a quantity of Na_2CO_3 equiv. to 7-8% of the $NaCN$ used).
- c. Filter off the ppt. immediately.
- d. add filtrate at once to 1 g. more of $ZnCl_2$ diss. in as little as possible of 50% $EtOH$.
- e. Filter off the $Zn(CN)_2$.
(Note must filter $Mg(OH)_2$ & $MgCO_3$ mixed to prevent formation of dark colored $Zn(CN)_2$)
- f. Wash $Zn(CN)_2$ with H_2O & then dry in air at $50^\circ C$.
- g. only imp. thing is to avoid an excess of $ZnCl_2$ over $NaCN$ otherwise get sticky mass.
- h. Product 91% $Zn(CN)_2$ + 10% - mostly $NaCl$ residue.



A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

Vanillin

NO. 01

Date: 10-20-47

By: H. G.

b. add 1 g of 0.4% d. in 1.5-2.0 ml of dry Et₂O.

c. add sufficient dry Zn (CN) equiv to 1.5 mol for every mol of phenol.

d. start a titration & pour in HCl rapidly till Zn (CN)₂ disappears with the formation of a milky sol (H₂O added).

e. more HCl dissolved & mixed above the sep. as a thick oil.

f. Takes 10-15 min to react with HCl.

g. & the porous HCl more slowly & continue to stir for 1 hr. more to be certain.

that all phenol has reacted.

h. pour Et₂O from solid & with the oil & dec. below.

fields

residual aldehyde 95%
p-nitrophenol aldehyde 85%

i. add 100 cc of 10% H₂O to inside. HCl & raise to b.p.

j. filter mixed & allow filtrate to cool → 50% yield

k. filter & allow filtrate to stand 10-15 hrs → 2.5% yield

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

Vanillin

Date: 11-11-47

By: 14-6

J. A. C. S. 416, 1518 (1944)

Radams & C. Montgomery

modifications in Lewis procedure

1. Using 96-98% KCN or MgCl_2 was added & the Zn (98%) was 95-98% pure.
2. This reaction is done in dry C_6H_6 (better stirring)
3. Use Zn (98%) equal to 2 mols for every mol of phenol or phenol ether.
4. Cool & attach unit.
5. Maintain 140° rapidly for 2 hrs.
6. Cool (& keep so), remove condenser & add 1.5 mols AlCl_3 .
7. Continue stirring & maintain 140° slowly at $40-45^\circ$ for 3-4 hrs.
8. Add 7" to an excess of 10% HCl which causes a heavy part of mixture HCl to sep.
9. Reflux for 2 hrs \rightarrow oily product the alcohol.
10. Then either
 - a. extract directly
 - b. filter from $\text{HCl}-\text{C}_6\text{H}_6$ mix.
 - c. steam distill & extract from distillate

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: Venilla

No. 6 vi
Date: 10-22-47

By: 1-1-6

11. $\text{NaCN} \rightarrow$ now yields (also KCN)

Chambers

1. amide

$\left. \begin{array}{l} 51.9 \text{ amide} \\ 57.9 \text{ Zn(CN)}_2 \\ 45.4 \text{ AlCl}_3 \\ 65.4 \text{ C}_6\text{H}_6 \end{array} \right\} \rightarrow 55.59 \text{ amide aldehyde}$
 (Quant.)
 s.p. ~ 46-47°C

- steam distill after decomp. with HCl
- Remove C_6H_6 & collect amide aldehyde & amides.
- Heb. with $\text{NaHCO}_3 \rightarrow$ addn product
- Dec. addn. prod. with NaOH distill

2. Thymol

$\left. \begin{array}{l} 51.9 \text{ thymol} \\ 57.9 \text{ Zn(CN)}_2 \\ 50.9 \text{ AlCl}_3 \\ 61.9 \text{ C}_6\text{H}_6 \end{array} \right\} \rightarrow 55.59 \text{ aldehyde}$
 (quantitative yield)

3. $\text{HO} \langle \text{C}_6\text{H}_5 \rangle \text{OCH}_3 \rightarrow \text{ald.}$

p-cresol methyl ether

$\left. \begin{array}{l} 51.9 \text{ H}_2 \langle \text{C}_6\text{H}_5 \rangle \text{OCH}_3 \\ 57.9 \text{ Zn(CN)}_2 \\ 45.4 \text{ AlCl}_3 \\ 75.9 \text{ C}_6\text{H}_6 \end{array} \right\}$

9-10-7-47

20-250-251°C

A. BROTHMAN & ASSOCIATES

No. 1 of

Date: 10-20-47

JOB:

By: H.B.

SUBJECT: Vanillin

amide

org. constituents 9, 10 (1949)
Hess & Hagen 70-71% yield

PCR → 2-chloro-CPD.

cl. should give 0-cl. deriv of amide

Antic

α-Naphthyl Thiourea

specific for R₂S

non-toxic to humans

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: _____

Vanillin

No. _____ of _____

Date: 12-11-47

By: H. G.

amyl acetate 100% Vanillin. Vol. 8, p. 815

1. carry out test by oxidation of amyl

2. 3 Kg. amyl (conty 90-95% amyl)

3. add 3.6 Kg. dichromate mixture

$\left\{ \begin{array}{l} 1 \text{ Kg. Na}_2\text{CO}_3 \\ 4 \text{ Kg. 25\% H}_2\text{SO}_4 \\ 1 \text{ Kg. conc. H}_2\text{SO}_4 \end{array} \right.$

at 55-60°C. & stir

4. Heat to 70°C

5. Cool.

6. Filter thru linen cloth

7. Extract with C_6H_6

8. Purify with NaHSO_3

9. Distill under vacuum.

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: Vanillin

Date: 10-31-47

By: H. G.

3. Name: 4-Hydroxy-Benzaldehyde

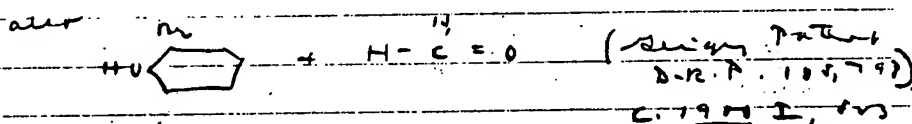
Ref. 2 (82)

1. Pass an 1 mol wt of the residue containing
cooling water, a cooled soln of 1 mol wt of
p-OH benzaldehyde ethyl ester C_4H_5O

2. Let stand for 2 days.

3. Crap the C_4H_5O on a boiling water bath
crystallizing the residue. When upon
the 3, 5 deriv. remains undissolved

mg, ref Ref. 28, 2409 (Paul)



Prop

capsule - from H_2O
m.p. 124.2

solubility: Et_2O , $EtOH$, $EtOAc$, $AcOH$,
 Me_2CO .

insolubility: CS_2 , CH_2Cl_2 , C_6H_6

27% sol CO_2H_2H & H_2O

more in cod.

JOB:

SUBJECT:

Vanillin

Date: 10-5-4

By: H-6

3-Cl-4-OH Benzaldehyde

ref. 2, 31

1. Dissolve (intermediate) into an ice-cooled CH_2Cl_2 soln of $\text{HI} \cdot \text{C}_6\text{H}_5$

orig. article

no. 37, 4030 (ref. 5)

Phys. Prop.

needles from H_2O

m.p. 159°C. b.p. 149-150°C

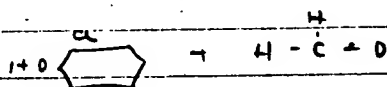
sol. easily sol. EtOH, Et₂O

diff. sol. CH_2Cl_2

insol. cold H_2O

air drying rates (D.R. 4054798)

C. 1900 I, 505



A. BROTHMAN & ASSOCIATES

No. 73 of

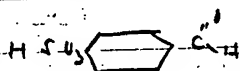
Date: 10-11-47

By: H. G.

JOB:

SUBJECT:

Vanillin



ref

oil: 11, 205

1. pour H_2SO_4 & CH_3 into a mix of 11-12
g 25% oil (with an excess of oil
and)

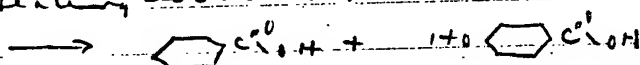
orig article

D.R.P. 154, 228

C. 1904 II, 1269

per b

1. starting with K_2H_4 140 H at 220-240



ref 39, 2511

2. Na salt \rightarrow needles

C. 1904 II, 1269

KL. 120.

27/4 (1910)

[14/10 (1904)

1. 1st dist H_2O in oil
2. then pour H_2O & CH_3

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: Vanillin

Date: 10-31-47

By: 146



orig. al

File 23, 2409 (1948) C. Paul

1. Dissolve p-OH benzaldehyde in 15 times the wt of CH₂Cl₂.
2. Cool the soln. (some add. stabilizer)
3. Pass in the theoretical amt of Br₂ (diss. in CH₂Cl₂). add in small portions under agitation & superficial cooling.
4. During the bromination a yellow oil sep. out which under the continued evolution of HBr passes into the form of a crystalline substance — the HO-C₆H₃(Br)-CHO. P. believes that the oil represents an addition product which then changes into the substitution product. i.e., let reaction mix. stand for 1 day to get a good yield.
5. Filter off the CH₂Cl₂ soln.
6. The filtrate contains a considerable amount of product & this is recovered from evap. of the CH₂Cl₂.
7. In order to completely exp. the CH₂Cl₂ a ~~transfer~~ product to a porous plate filter & wash with a small amt of H₂O.

A. BROTHMAN & ASSOCIATES

JOB: _____

Date: 10-31-47

By: 17-6

SUBJECT: Vanillin

- trial oil H.M. is used.
 c. V. stilling several times from boiling H.M.
 2. The yield is good.

See 23, -411

c. Paul

on the assumption that the Me
 chain will easily be substituted then
 the aldehyde by ~~CH₃CH₂CH₂~~ CH_3 , P. tested
 the $\text{H}_2\text{O} \begin{array}{c} \text{Me} \\ \diagup \\ \text{C} \\ \diagdown \end{array} \text{C}^{\text{H}} \text{H}$ with NaOCH_3 in a
 sealed tube. The reaction takes place
 only at elevated temp & the main yield
 of product is a badly decomposed
 mass. only traces of Vanillin could
 be found.

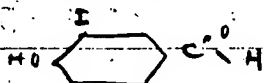
A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: Vanillin

Date: 10-31-42

By: H. G.

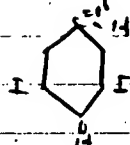


Ref. Publ. 3, 93 Ent. No. 746

Ref. 23, 2413 (1895) C. Paal.

1. Thyphlo { ann. d. chm. 146, 294 }

res. a derivative was the



mono-I deriv. H. used

1. (P. 11) p-OH ϕ CHO

2. (P. 11) I₂ (= mol)

3. P. 11 etc.

4. P. 11 H₂O

but at equimolar parts of p-OH and I are used then only 1 of der quant is obtained

2. Fabry 11 above in (the ~~same~~ same same)

is obtained a small quantity of mono-I-deriv

m. p. 127°C

see in etc. & A. H. 14

first dis under of p-OH old in A. H. 14 when in considerable quant

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: _____

Date: 11-31-47

By: 17-6

Paul obtains Di I from by using
 6 parts p-OH cool.
 1.5 parts I
 1.1 part HI
 70 parts oil
 1.5 parts 17014

Δ under reflux
 +

14.4 ClC1=CC=CC=C1 C₁₄ obtained was not prepared
 by P. in a multistep - he had
 trouble. Yield 90% 51.21
 Found 49.73

no mention is made of forming Vanillin
 from Oc1ccccc1 C₁₄

14.4 ClC1=CC=CC=C1 C₁₄ see 37, 407 (1904) Warty
 1. 100 g of p-OH & C₁₄ in 500 ml H₂O
 warm & then cool. Filter off solids
 wash & cool again (Cis?)
 2. 100 g of oil & 1 lb of the oil obtained
 from 3.7 g of 17014 & conc. HCl

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

Vanillin

Date: 10-21-47

By: H.B.

(This is a modification of the —
 Evans et al. → dist. dist.)
 the vacuum to exhaust it from
 reaction flask.

3. Extract for a short time & distill
 /C H₂ from a water bath.

4. Stillhead removed from a small end
 of etc. → m.p. 154-156°C.

5. Stillhead removed = m.p. 149-151°C
 (at 14 mm)

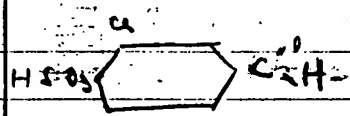
6. Stillhead from H₂O/H → 159°C m.p.
 (on long distillation)
 fine white needles.

OTHMAN & ASSOCIATES

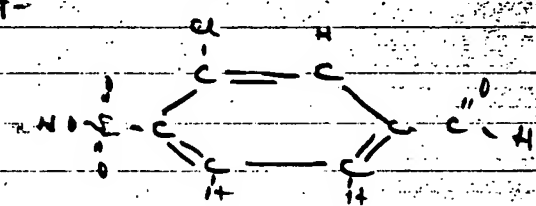
Date: _____

By: _____

SUBJECT: Vanillin



Cont. No. 1572



$\text{C}_7\text{H}_8\text{O}_4.5\text{Cl}$

Don't mix with

Vanillin

A. BROTHMAN & ASSOCIATES

JOB: _____

SUBJECT: Vanillin

Date: 12-31-47

By: 17-6

rec 39, 2511 (1906) c. ^H pure, 100%

1. 10 g. ~~NaOH~~ c1ccccc1 ^{C¹⁸} _H (9.22)

2. a. with 50 g. KOH & 10 g. H₂O. @
220°-240°

3. yield 53 g. ^{2.1 g.} c1ccccc1 ^{C¹⁸} _H — p. 113-114

3.29. 141 & C¹⁸_H — p. 203°C
4020 25476

A. BROTHMAN & ASSOCIATES

JOB:

Date: 10-31-47

By:

SUBJECT:

Vanillin

Vanillin oil. Cpt No. 773 9, 248.
 Cere oil. Cpt No. 52657 6, 399.
 p-Cere oil 6, 399.

NaOH



NaOH

12 NaOH

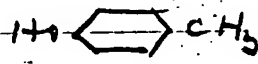
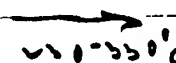
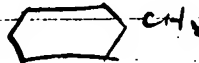
oil 6, 399

Sample 1

ng. cum. 2 (1943)

NaOH

NaOH



ye

TO: 100-2

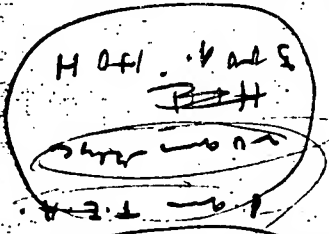
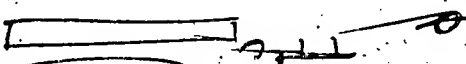
~~~~~



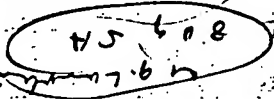
in complete  
mixture

element

CH<sub>3</sub> - CH<sub>2</sub>CH<sub>2</sub>OH



~~1000~~



①

6/6/50  
me



# A. BROCHMAN & ASSOCIATES ( )

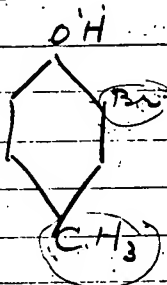
JOB: \_\_\_\_\_

SUBJECT: \_\_\_\_\_

No. \_\_\_\_\_ of \_\_\_\_\_

Date: \_\_\_\_\_

By: \_\_\_\_\_



#2

JOB: \_\_\_\_\_

SUBJECT: \_\_\_\_\_

- ✓ 1. Peacock
- ✓ 2. Blue catalyst
- ✓ 3. Facial
- ✓ 4. Vanish Cream &c
- ✓ 5. Extraction (Send for sample)
6. methyl methacrylate Synthesis
7. Patent Work on methyl methacrylate

~~A. Conclusion~~

B. Set

Twelve  
Twist Ball  
Santopom  
Nacronal - natural alumin  
Diponal - alcohol curable  
acrylic resins

Santopom  
Paraplow

~~C. Idea of Rough Surface (by Chipping)~~

D. Take removed paper & coat with  
dyeing.

E. mechanical means of applying  
rule & Powder

# A. BROTHMAN & ASSOCIATES ○

JOB: \_\_\_\_\_

SUBJECT: \_\_\_\_\_

No. \_\_\_\_\_ of \_\_\_\_\_

Date: \_\_\_\_\_

By: \_\_\_\_\_

## 2. Blue Catalyst

- ~~a. catalyst (nil)~~
- ~~b. water based paint (nil)~~
- ~~c. water based paint~~
- ~~d. water based paint~~

## 3. Paschal

- ~~a. Finish Sausage~~
- ~~b. White Report~~
- ~~c. Water based paint~~

## 4. Vanishing Cream

- a. oil {
  - alcohol
  - carbowax
  - propylene glycol
  - carbide
  - menthol

b. Prep. Sample for Control

c. Prep. Sample for "Tong"



200 g  
500 g

1119  
Add

117  
156

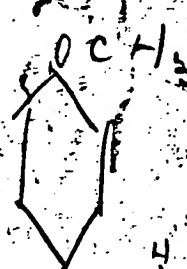
159

153  
107

108  
44

365  
10

135 10 90



365 x 60 =  
108

20 g HCl

115 cc 38  
10

6/6/50  
2

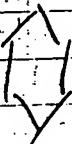
# A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

Date:

By:

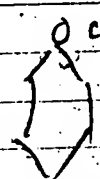


2 x 19.0 x 5.0  
1.36.3

13.6.3 x 1.10 = 139.0 gms  
2 x 49.0 2 x 2.0

139.8 x 1.12 = 156.6 gms  
0.98 2 x 2.0  
150.9 NACH + 12.0 cc H<sub>2</sub>O

1.10 / 137 = 0.008  
31.08 5.0  
150.9  
102.0 cc C<sub>6</sub>H<sub>6</sub>



0.37 x 1.5 = 0.555  
117.00 2 x 1.8 gms



#3

SUBJECT: Chloroform

Ref. Thorpe Vol III p 09

1. prep. by reduction of  $\text{CCl}_4$  with nascent Hydrogen.
2. method — stir a mixture of  $\text{CCl}_4$  and finely divided Fe in water; the reaction is initiated by the addition of a small amt. of  $\text{HCl}$ .

U.S. Patents } 753,125  
                  } 1,107,025.

Ref. Ullmann

1. can control reaction (above) by exerting stirring & keep any  $\text{CHCl}_3$  from forming. Thomas is used in the U.S.

2. also. C.7. 586,006

1700 g.  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$

200 cc  $\text{H}_2\text{O}$

a. add 1750 cc 30%  $\text{NaOH}$   
b. 500 cc  $\text{CCl}_4$

c. warm gradually under stirring till  
b.p. of  $\text{CHCl}_3$  is reached.

d. yield 90%.

# A. BROTHMAN & ASSOCIATES

JOB: \_\_\_\_\_

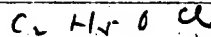
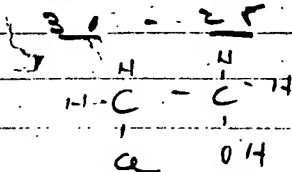
SUBJECT: \_\_\_\_\_

No. \_\_\_\_\_ of \_\_\_\_\_

Date: \_\_\_\_\_

By: \_\_\_\_\_

ans. complete



rel I 3-4

rel E II 3-4

2.4

$\begin{array}{r} 1719 \\ 1944 \\ 2554 \\ 2842 \\ 3091 \end{array}$

$\begin{array}{r} 2858 \\ 2843 \\ 3089 \\ 4122 \end{array}$

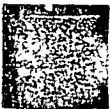
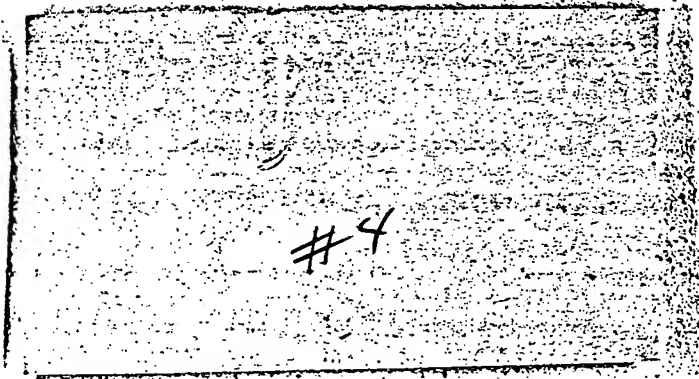
4306

935

1476

2.3

$\begin{array}{r} 3778 \\ 3059 \\ 3547 \\ 4028 \end{array}$



# A. BROTHMAN & ASSOCIATES

JOB: \_\_\_\_\_

SUBJECT: \_\_\_\_\_

Date: \_\_\_\_\_

By: \_\_\_\_\_

Exner & Amund

✓ 9-191-5 1 ply (200cc) 6 mm. Carlisle #3.25

✓ 14-097- Rubber gloves, Niprene, heavyweight Size 9 1.40

✓ 14-836-1 Alkaid test paper, box of 100 1.00

✓ 15-565- 3. Brush, arrow joint 0.75

✓ 3-636 1 doz. Brush, tube, soft bristle 0.75

✓ 11-162 1- Flowmeter, pyrex 6.80

✓ Par. C. Chen # E-139 1 set

acid H<sub>2</sub>CO<sub>3</sub> # A-300 2 m

✓ 100 Natto 100 c.p. 14 6 m.

✓ 100 # 5-233

✓ 100 CaCO<sub>3</sub> porta # C-162

✓ # 2-120 Malware Air

End of list

# A. BROTHMAN & ASSOCIATES

JOB: \_\_\_\_\_

SUBJECT: \_\_\_\_\_

Date: \_\_\_\_\_

By: \_\_\_\_\_

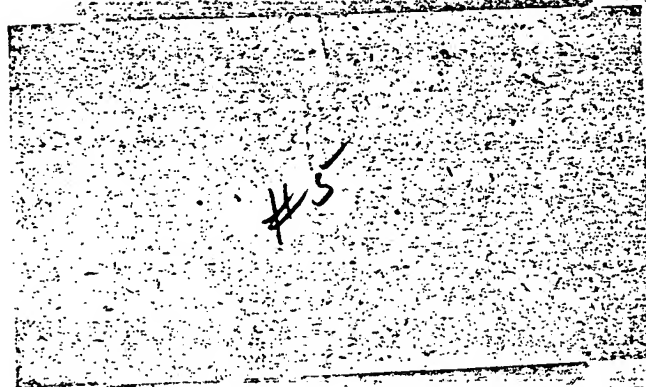
S & A

|                  |               |                                            |                  |
|------------------|---------------|--------------------------------------------|------------------|
| <del>17520</del> | <del>1</del>  | <del>dy. rubber bulb for dropping</del>    | <del>0.15</del>  |
| <del>17525</del> | <del>3</del>  | <del>20</del>                              | <del>0.50</del>  |
| <del>2</del>     | <del>30</del> | <del>"</del>                               | <del>0.50</del>  |
| <del>17805</del> | <del>2</del>  | <del>rubber bulb for pressure only</del>   | <del>0.60</del>  |
| <del>17810</del> | <del>1</del>  | <del>for vacuum or pressure</del>          | <del>0.60</del>  |
| <del>17800</del> | <del>2</del>  | <del>Enticon ring, 3/8" H.</del>           | <del>0.50</del>  |
| <del>3</del>     | <del>"</del>  | <del>"</del>                               | <del>0.50</del>  |
| <del>17790</del> | <del>2</del>  | <del>rubber rings, 3" O.D.</del>           | <del>0.54</del>  |
| <del>3</del>     | <del>"</del>  | <del>4 1/4" O.D.</del>                     | <del>0.54</del>  |
| <del>2</del>     | <del>"</del>  | <del>5 1/2" O.D.</del>                     | <del>0.80</del>  |
| <del>17785</del> | <del>24</del> | <del>test tubes, pyrex 150 x 11 mm.</del>  | <del>1.108</del> |
| <del>12</del>    | <del>"</del>  | <del>175 x 22 "</del>                      | <del>0.28</del>  |
| <del>17145</del> | <del>6</del>  | <del>48 "</del>                            | <del>0.50</del>  |
| <del>17405</del> | <del>3</del>  | <del>pyrex funnel 60°, 50 mm diam.</del>   | <del>0.81</del>  |
| <del>3</del>     | <del>"</del>  | <del>" " 75 " "</del>                      | <del>1.02</del>  |
| <del>3</del>     | <del>"</del>  | <del>" " 100 " "</del>                     | <del>1.25</del>  |
| <del>17105</del> | <del>2</del>  | <del>pyrex funnel flasks 50 mm diam.</del> | <del>0.44</del>  |
| <del>2</del>     | <del>"</del>  | <del>100 mm.</del>                         | <del>0.22</del>  |

Glass Richter funnels.

|                 |                |                                     |                 |
|-----------------|----------------|-------------------------------------|-----------------|
| <del>1220</del> | <del>6</del>   | <del>Griffin beakers, 600 cc.</del> | <del>1.50</del> |
| <del>2010</del> | <del>1/2</del> | <del>100 mm. plate</del>            | <del>0.70</del> |
| <del>2010</del> | <del>1/2</del> | <del>"</del>                        | <del>0.70</del> |
| <del>2010</del> | <del>1/2</del> | <del>"</del>                        | <del>0.70</del> |





22 Aug 47

A. BROTHMAN & ASSOCIATES

Chemical and Mechanical Engineers

114 EAST 32nd STREET

NEW YORK 16, N. Y.

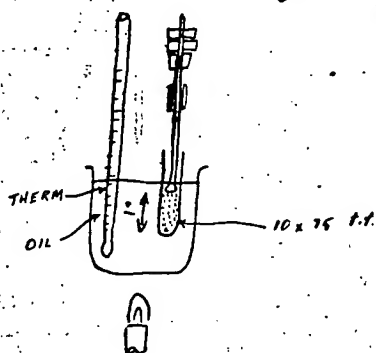
Flow point of ground Lot 54 molding powder

13 x 100 t.t.

0.145" plunger, 373 g. wts.

| <u>Time</u> | <u>Temp</u> | <u>Remarks</u>                           |
|-------------|-------------|------------------------------------------|
| 4:10 PM     | 125°C       | Beginning of const. temp. period         |
| 4:25 PM     | 125°C       | Insert plunger & begin heating           |
| :28         | 128°C       | 1/4" fall on putting weights on plunger. |
| :32         | 132°C       | 1/8" fall                                |
|             |             | 1/4" fall                                |
| :39         | 138°C       | 3/8" fall                                |
| :46         | 146°C       | 1/2" fall                                |
| :53         | 153°C       | 5/8" fall                                |

## Standardization of molting powder test.



Heating: Heat rapidly to  $120^{\circ}\text{C}$ .

Hold 10 min.

Heat @  $1^{\circ}/\text{min}$  to flow point

Flow point taken as point at which plunger has sunk  $\frac{5}{8}$ "

Results:

Ground R & H injection powder:  $149^{\circ}\text{C}$

Lot No. 54, ground:  $186^{\circ}\text{C}$

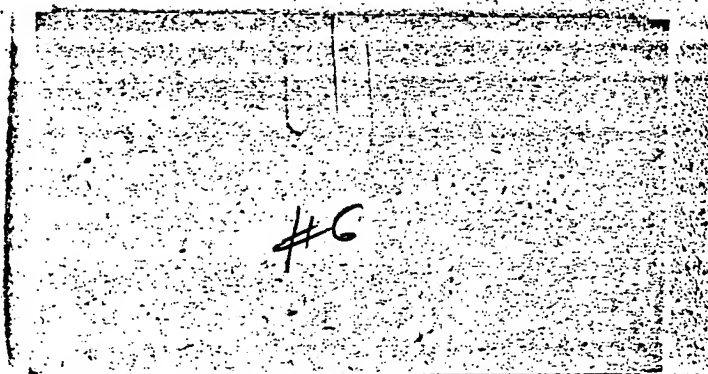
R & H compression powder: no flow point. Originally plunger sank, then rose again.

The apparatus was then modified as follows:

- 1) A  $18 \times 750$  test tube substituted for  $10 \times 75$
- 2) This tube filled initially to  $11\frac{1}{2}$ "
- 3) Flange on plunger eliminated. Plunger  $0.145$ " diam. Plunger + weights =  $373\text{ g}$ .
- 4) Plunger only put in after material is at  $120^{\circ}$  & tacky enough to prevent sliding in of plunger without compression of particles. Flow point ( $\frac{5}{8}$ " plunger sinking) is measured from 30 sec after plunger inserted.

Results: R & H comp. powder:  $157^{\circ}\text{C}$ ,  $160^{\circ}\text{C}$ ,  $166^{\circ}\text{C}$  (last sample trapped too much originally)

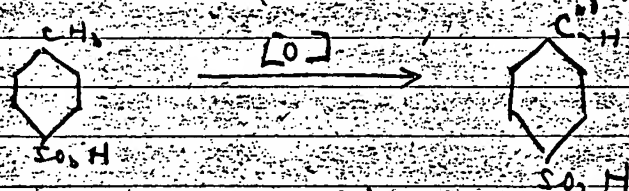
Lot No. 54:  $148^{\circ}\text{C}$ . Is this due to larger particle size than R & H or to lower P.T.?



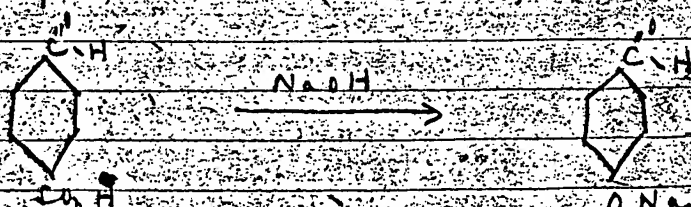
## Work to be Done

It was decided to investigate the feasibility of carrying out the following general reactions with a view to producing certain substituted aromatic aldehydes:

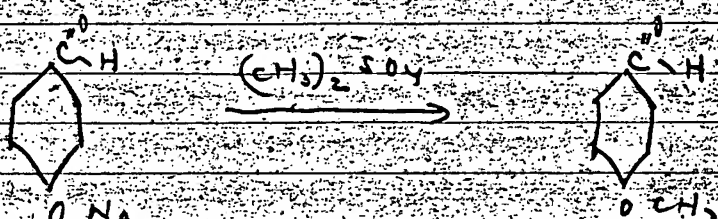
1. The oxidation of a methyl group (present in the para position on a ring to a sulfone and radical) to an aldehyde structure, i.e.,



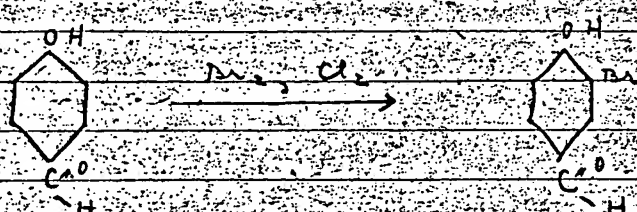
2. The replacement of a sulfonic acid group (present in the para position on a ring to an aldehyde group) by an  $\text{OH}$  linkage, i.e.,



(present in the para position on a ring to an aldehyde group) to an alkyl ether structure, i.e.,

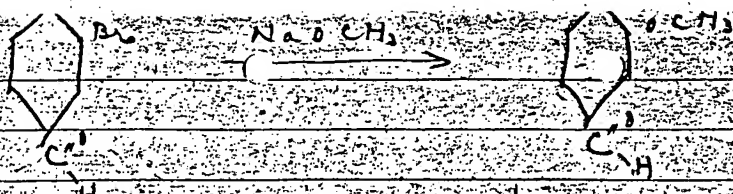


4. The halogenation of an aromatic hydroxy aldehyde (with the OH para to the  $\text{C}_6\text{H}_5$  group) to yield a monohalogenated derivative with the halogen substituted meta to the OH group, i.e.,



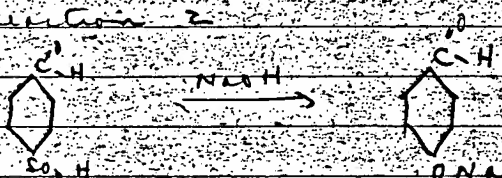
5. The replacement of a halogen group on a ring (present ortho to an OH group and meta to a  $\text{C}_6\text{H}_5$  group) by an alkyl group (R = alkyl group), i.e.,



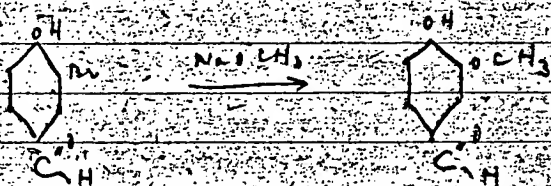


The basis for a successful accomplishment of the above work has been thoroughly gone into, both from the standpoint of a chemical literature search (and beyond laboratory work). The results may be summarized thus:

1. Reaction 2



and Reaction 5



cannot be carried out to give any significant yield of the products desired.

2. Reactions 1, 3, and 4 are practical

for all conditions involved.

3. an alternative method for the production of anisaldehyde is given.
4. The proposed course of any further work directs toward a successful solution of the problem of a low cost method for the production of substituted aromatic aldehydes is detailed.

## I. Investigation of Individual Reactions

A. oxidation of *p*-Toluenesulfonic acid  
to *p*-Benzaldehyde sulfonic acid -

Two methods are described in the literature for the <sup>removal of the</sup> methyl group <sup>in any</sup>

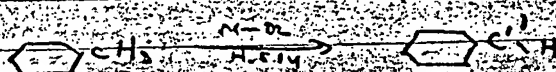
to the aldehyde structure by means  
of the cheap and readily available  
 $MnO_2$  (pyrochite)

Ref. ① *J. Chemical Industry of Russia*

Vol. 6, p. 1439-1440 (1929)

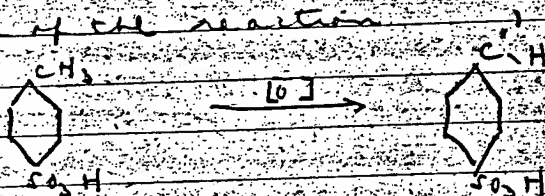
T. S. Shenderovich and S. S. Livshits  
(original article)

*Russian Chemical Abstracts* 1936, 92



2. At 70° favors the formation of tar, while below 55° C, the reaction is slow and more benzoic acid is formed.

3. An excess of pyrosulfite may cause the formation of tar during the distillation with steam (this problem would not be encountered in the case of the reaction)



as the sulfonic aldehyde produced is soluble in water.)

4. Prolongation of the reaction tends to increase the relative quantities of benzoic acid and tar.

Ref. D. German Patent 154,527

Kl. 130

applied for April 27, 1902


granted October 14, 1904

(original article)

Deutscher 11, p. 223

Chemische Zentralblatt 1904, II, p. 126

1. First dissolve  $MnO_2$  in  $95\%$   $H_2CO_4$  - use an excess of the acid.

2. Then pass in the  $H_2SO_4$    $CH_3$

### Conclusion

1. The method of the diacid and diacid would seem to be the one most likely to work successfully. It does not seem as though the fact that the oxidation here was carried out in toluene would give any different results were the p-toluenesulfonic acid used.

2. The Simons Patent appears to have the flaw in it that the use of  $2.5\%$  solution might very likely result in further sulfonation. However, this might be true.

a.  $MnO_2$  dissolves in  $95\%$   $H_2CO_4$  and in oleum with the formation of (a green colored) manganese heptoxide  $Mn_2O_7$  which very readily gives up its oxygen even at low temperatures.

b.  $Co_2$  by the use of the proper time and temperature conditions as may be possible to oxidize the  $CH_3$  group and separate the product before any appreciable sulfonation can

3. no yields are given for either reaction though the original articles may state yields.

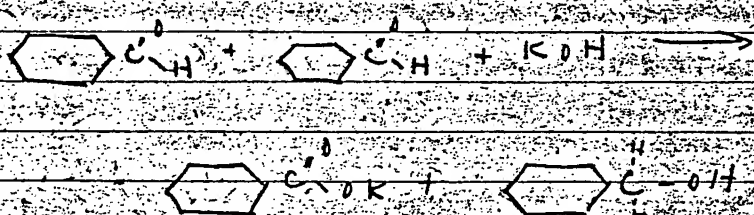
D. The conversion of p-benzaldehyde sulfonic acid to p-benzylidene benzaldehyde.

literature ref D organic chemistry by

Sakaguchi 1 Frank W. Whitmore

p. 791

1. alcohols cause the Cannizzaro reaction to take place with an aromatic aldehyde



2. If the aldehyde is entirely peroxide free, the reaction does not take place (Kharasch 1935)

ref D Berichte 39, 2511 (1906)

C. Frank and H. Kraft

1. Take 10 gms. of  $\text{Na}_2\text{SO}_3$   $\text{C}_6\text{H}_5\text{CHO}$  (92%)  
this is the technical grade. A & K.



2. Used 5.0 g of K<sub>2</sub>H and 10 g of H<sub>2</sub>O<sub>2</sub>  
at 220-240°C.

3. yield (total) - 5.9 gms of product, which  
on distillation  
gives 2.1 gms. C1=CCCCC1C(=O)O 46% of theoretical

4. 3.3 gms. O=C1C=CC(=O)OCC1 57% of theoretical

There should have been obtained

$$\frac{\text{C}_6\text{H}_5\text{C}(=\text{O})\text{H}}{\text{NaOH} \cdot \text{C}_6\text{H}_5\text{C}(=\text{O})\text{H}} \times 9.2 = \frac{122}{194} \times 9.2 = 5.8 \text{ gms.}$$

$$\frac{\text{HO} \cdot \text{C}_6\text{H}_4\text{C}(=\text{O})\text{H}}{\text{NaOH} \cdot \text{C}_6\text{H}_4\text{C}(=\text{O})\text{H}} \times 9.2 = \frac{138}{194} \times 9.2 = 6.5 \text{ gms.}$$

Thus the losses entailed due to decomposition are considerable.

### Conclusion

1. The work of Smith and Krieger shows



group on (an aromatic) aldehyde is not a feasible one due to the extreme conditions required and under which the  $\text{C}^{\alpha}\text{H}$  group either

- a. is oxidized completely to  $\text{C}^{\alpha}\text{O}$
- b. or is decomposed entirely.

2. The work of Kharasch should be looked up to see whether by elimination of the peroxide from the aldehyde, the tendency of the  $\text{HC}_6\text{H}_4\text{C}^{\alpha}\text{H}$  to undergo a Cannizzaro reaction can be eliminated.

3. The formation of anisaldehyde from p-Hydroxy Benzaldehyde.

Literature The reference given below refers to the preparation of anisole from phenol but it most likely can be used to prepare anisaldehyde from



6. then and 5. then

1. add 25.5 gms of phenol (2.5 mols)  
and 100 gms of NaOH (2.5 mols) to  
1000 cc. of water.

2. Cool to below  $10^{\circ}\text{C}$ .

3. Then add 31.5 gms (2.5 mols) of  
 $(\text{CH}_3)_2\text{SO}_4$  over a period of 15 min.  
The reaction must be kept  
bet.  $20-25^{\circ}\text{C}$  all of this time.

4. Heat the mixture to  $100^{\circ}\text{C}$  for a  
 $\frac{1}{2}$  hour.

5. Then add 2.5 mols more of phenol  
over a 15 minute period.

6. Then reflux for 15 hrs.

7. Cool the mixture.

8. Separate the amide layer.

9. Extract the water layer with  
benzene and add it to the amide.

10. Strip off the benzene.

11. Distill the amide under vacuum  
(bp.  $153-154^{\circ}\text{C}$  at 748 mm).

12. Yield (based on dimethyl sulfate)  
= 75%.

density 2.42 cd.

(Sattermann and Willand  
p. 244)

1. dissolve 0.2 mol (19 gms) of phenol in  
100 cc of 2N NaOH

2. add 26 gms of  $(CH_3)_2SO_4$  in  
3 portions over 5 minute intervals  
and under agitation

3. Test to see if the aqueous solution  
on which the anisole floats is  
no longer acid.

4. add 20 cc of 2N NaOH and heat  
at  $100^\circ C$  for  $\frac{1}{2}$  hour to complete  
the reaction and destroy any  
 $(CH_3)_2SO_4$

5. Cool and remove the aqueous layer

6. Dry the anisole and distill

7. yield (based on phenol) = 90.7%

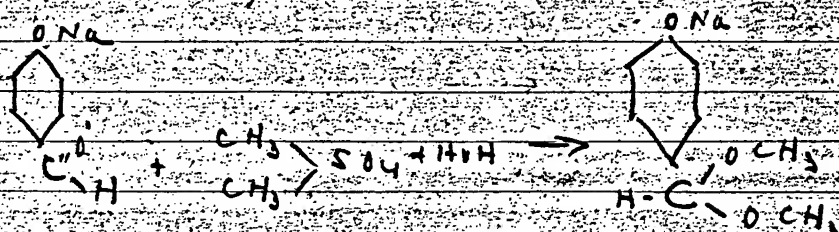
### Evaluation

1. The method of Huns and Hager  
utilizing acid does both  $CH_3$   
groups of dimethyl sulfate available  
to be the more logical choice

2. The technique would probably not

choose the product anisaldehyde boils at  $247^{\circ}\text{C}$  as against  $154^{\circ}\text{C}$  for anisal.

The only drawback to the process may be the possible formation of aromatic acetals.



but this reaction does not seem very likely as it is not mentioned in Whitmore's, Vortman von Richter's and Meyer-Jacobson's texts. Still a further search should be made in Reineken and the more recent literature. Very likely, however, the conditions for the reaction could be set as to hydrolyze the acetal as fast as it is formed.

The preparation of 3-Mono-4-Hydroxy-Benzaldehyde  
from p-Hydroxybenzaldehyde  
literature - reduction data for the synthesis  
background of both the Br and Cl derivatives of p-Hydroxybenzaldehyde are given.

C. Part

1. Dissolve p-Hydroxy benzaldehyde in 15 times its weight of warm  $\text{CHCl}_3$ .
2. Cool the solution (some aldehyde crystallizes out).
3. Add the theoretical amount of  $\text{Br}_2$  (dissolved in  $\text{CHCl}_3$ ). The addition should in small portions and under agitation and some cooling.
4. During the bromination a yellow oil separates out which, under the continued evolution of  $\text{HBr}$ , passes into the form of a crystalline substance - the  $\text{HO}-\text{C}_6\text{H}_4-\text{C}(\text{H})=\text{CH}_2$ . P. believes that the oil represents an addition product which then changes into the substitution product. Therefore, the reaction mixture should be let stand for  $\frac{1}{2}$  day in order to get a good yield.
5. Filter off the  $\text{CHCl}_3$  solution.
6. The filtrate contains a considerable quantity of product and this is recovered on the evaporation of the  $\text{CHCl}_3$ .
7. Add the residue from the  $\text{CHCl}_3$ .



to the main portion of product and wash under vacuum with a small amount of <sup>old</sup> water till all of the HCl is separated.

8. Crystallize several times from boiling water.

9. The yield is quantitative.

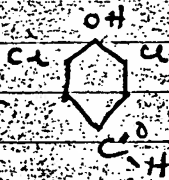
Ref. (3) Preparation of Oc1ccc(C=O)cc1

Berichte 37, p. 4032 (1904)

H. Biltz

1. Dissolve 3 gms of p-Hydroxybenzaldehyde in 300 gms of warm CHCl3. Filter off whatever crystals appear and cool again.

2. Then pass in over a one hour period the Cl2 obtained from 3.7 gms of K2CrO7 and conc. HCl (this is somewhat less than the theoretical amount; excess Cl2 would yield



) Use a slight vacuum

to exhaust the Cl2 from the evolution flask and to pull it into the reaction flask.



the CH<sub>3</sub> from a water bath.

4. Crystallize the residue from a small amount of alcohol to yield a material with an m.p. of 134-136°.
5. Distill the material under vacuum — take the fraction boiling at 149-150° at 14 mm.
6. Again crystallize the product from water. Fine white needles result. m.p. 139°.
7. No yield is given.

### Evaluation

1. The preparation of the bromine derivative would seem to be the best choice in spite of the relatively high price of Br<sub>2</sub> (21 cents/lb.) because the replacement of the CH<sub>3</sub> by an OCH<sub>3</sub> should take place far more readily than in the case of the Cl compound.

2. Reaction of 2-methoxy-*p*-cresol with bromine to yield Vanillin.

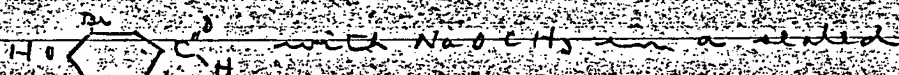
Structure — The references given herein only depend only refer to the direct preparation of vanillin from the methyl

... of the ...  
halogen atom on a ring.

410 Berichte 22, 2411 (1895)

C. Paal

on the assumption that the Br atom  
could more easily be substituted than the  
aldehyde group by  $\text{NaOCH}_3$ , Paal heated



tube. The reaction takes place only at  
elevated temperatures and the principal  
yield of product is a badly decom-  
posed mass. Only traces of vanillin  
could be found.

411 Chemiker Zentralblatt 1898 I, p. 432

Vanderhoff

and Liebig's Annalen der Chem. 133, p. 49

Fittig

with heating with  $\text{H}_2$  or  $\text{KOH}$  (even  
for 6 hrs) produces the slightest change  
in  $\text{C}_6\text{H}_5\text{Cl}$

412 Comptes rendus 53, p. 599

Liebig's Annalen der Chemie 131, p. 359

Richer

heating  $\text{C}_6\text{H}_5\text{Br}$  for several days with

Expt ④ Nitrobenzene 5 (2-chlorobenzene), p. 130  
Simon Patent 411,052 (Agfa)

when c1ccccc1[N+](=O)[O-] is heated with NaOH  
and MeOH at

2.5 atmos

190-200°C

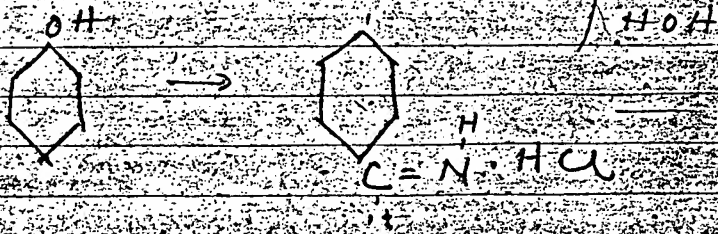
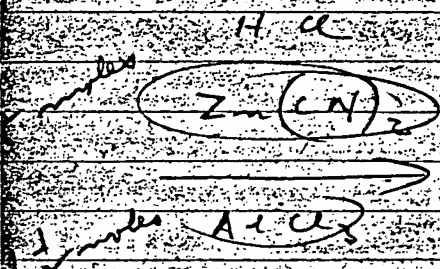
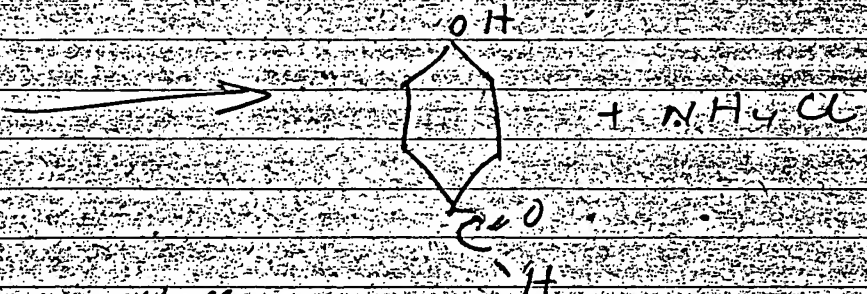
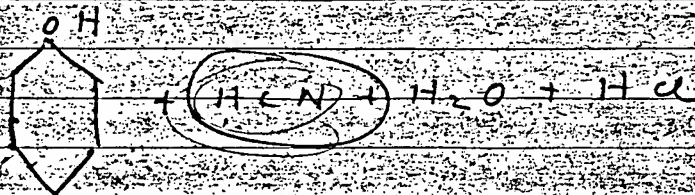
for 2-4 hrs.

a mixed product of anisole and  
phenol is produced.

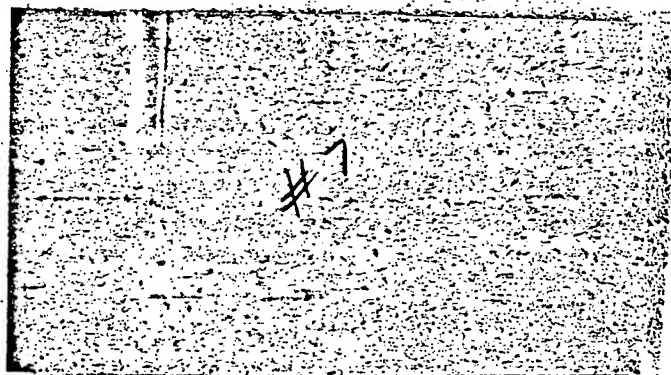
### Conclusion

1.

# Battermann Reaction



amide - hydrochloride





JULIAN PAUL BRODIE

September 13, 1946

Mr. A. Brothman  
A. Brothman and Associates  
114 East 32nd Street  
New York 16, N. Y.

Dear Mr. Brothman:

This will confirm the understanding in our conversation of August 29, 1946. It is understood and agreed that your organization will undertake to develop for me a men's vanishing cream, subject to the conditions agreed upon between us. It is understood and agreed that your estimate for the work involved will bear a direct total cost of \$1200 to \$1300, which will be defrayed by me.

It is further understood and agreed that you will incorporate into the finished product ingredients which will have genuine therapeutic effect on the skin.

It is further understood and agreed that the product you submit as final will be tinted a shade subject to my approval, scented with an odor subject to my approval, and will in no wise be irritating or harmful to the skin. A further, and indispensable property of the finished product you develop will be that its application to the skin will leave a slight residue of tan coloring on the skin of the user. Moreover, it is understood and agreed that the final product you submit for my approval must be commercially practical in the sense that it must not be composed of ingredients so costly as to make the final product unmarketable, and that the final product will be commercially possible to compound in quantity, to package in jars, and that it will retain its moisture and its properties for indefinite periods and will not depreciate or lose its efficacy while in packages on dealers' shelves.

In consideration of the fee agreed upon, it is understood that:

- (1) In lieu of any profit realized by you at this time, you shall receive a stock interest in any company which may be formed to produce and market the product.
- (2) In the event that the further development of this undertaking should involve the building of plant facilities for the manufacture of the cream, your firm will be given priority consideration in the engineering and designing of the plant.
- (3) In the event that any special trade secrets or patents shall develop or seem necessary during this work, you shall, for my interest, protect the same



JULIAN PAUL BRODIE

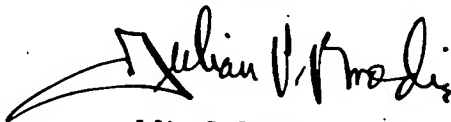
-2-

to the utmost extent. But it is understood that such secrets and patents shall become my property and assignable by me, at terms within my discretion, to the company which may be formed to exploit this product. (4) It is understood that you will take all steps to the end that the finished product will comply in all respects with such pure food and drug standards as are presently established by law. (5) It is further understood that you are to keep me continuously advised of the progress and development of your work and (6) It is understood and agreed that you will begin immediately on this work, that you undertake to complete it within three months or less and that you will deliver to me a reasonable quantity of your final product for test, consumer study and analysis.

It is understood that you will begin work as soon as this contract is executed by both parties and your signature below constitutes your acknowledgment of the receipt of a retainer payment in the amount of \$650. against the total sum named above. The balance will be payable at the time the project is completed in accordance with the specifications of this letter.

I look forward with great interest to the result of your work and assure you of my confidence that you will develop a satisfactory product.

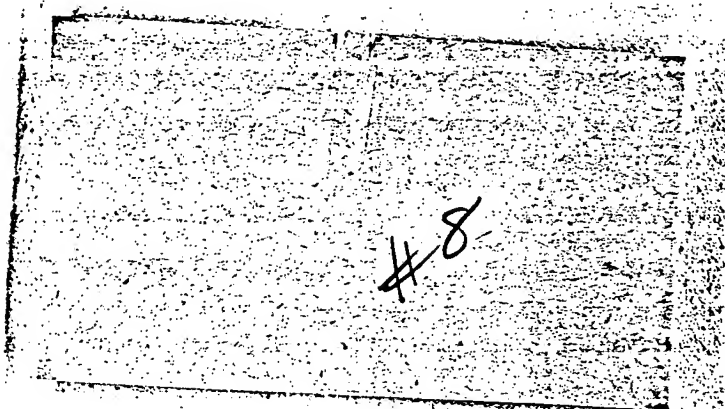
Very truly yours,



Julian P. Brodie

SIGNED AND AGREED TO THIS  
19 DAY OF September 1946.

  
A. Brothman



Analysis

(Titrated)

from ref. method - org. Reagents in  
long analysis

p. 417 H

I - Thiocyanate and

and detectable 0.001 mg Fe in a cone of  
1:10, 100, 1000

and determinable 0.0125 to 0.025 mg Fe

Interfering substances As, Bi, cyanides, Co, Au,  
Pb, Mn,  $Hg^{+}$ , molybdates,  
Ni, Ag,  $Cr^{+}$ , sulfites,  
W, thallium, Zn.

Reagents

- add 4 cc of  $HSCN$ ,  $COH$  to 8 cc of  $NH_4OH$   
in 50 cc of water
- perchloric acid
- $HClO_4$  - conc 4.105
- $NNH_3$  - conc
- $NH_4OH$  - c.p. 2.75
- distilled 1 gm pure Fe in dil  $HClO_4$  and  
 $HNO_3$ , with small oxide of  $N$  or  $NNH_3$  &  
dil to 1 liter

## Method

1. Add 5 cc sample with 5 cc of  $H_2SO_4$  and 0.5 cc 60% peroxide and mix well. Heat for 10 min, cool, & repeat 0.5 cc addition of peroxide and until color is colorless & free from excess peroxide and
2. Transfer to beaker & make alk to detours by addition of  $NH_4OH$  & then acid to detours with conc.  $HClO_4$  add excess of 5 drops of conc.  $H_2SO_4$
3. Add 1 cc of Thionylchloride and heat & cool. Add  $NH_4OH$  & adjust pH to color C. with standard.

Washed  
down

## Further

p. 156

millen

## C. Form

Na - yellow

Co - yellow red

Cyanide - deep orange - yellow

Am - amber

C (Ni) & ions - Red

p. h. - yellow to amber

Nm - amber

Hg - black

Mg - yellow - orange

NH<sub>4</sub><sup>+</sup> - reddish purple

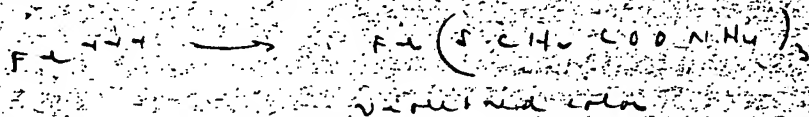
W - blue ( )

Ag - colorless

Cr<sup>3+</sup> - yellow

Uranium - yellow

Barium & Cobalt microanalysis actn 2, 257 (1957)



Fe<sup>3+</sup> does not give this reaction.

Fe<sup>3+</sup> not Fe<sup>3+</sup> Cd 2 p 97  
"extract"

2, 2' dimethyl



→ soluble deep red complex



Fe<sup>3+</sup> does not react  
Test useful unless CN<sup>-</sup> present

limit 0.05 p 72

con limit 1.1, 1.66, 200

reagents: 1. 70 vol % 2, 2' dimethyl in dil HCl  
or in the soln of the base

method - treat a drop of the test soln with a  
drop of the reagent soln on a dry plate  
→ red or pink color

KCNs used for Fe

presence of phosphates, arsenates, oxalates, tartrates & other org. O.H. acids, fluorides & many acids which give stable complexes  $Fe^{++}$  salts can so reduce the conc. of  $Fe^{++}$  that the maximum amount of iron for spect. is never reached.

$Hg^{+}$  &  $Hg^{++}$  also interfere

$NO_2^{+}$  interferes

Limit of Detect. 0.25  $\mu$  Fe

Concn. Limit 15,000,000

Method - mix a d.p. of the test soln with a drop of 1% KCN soln and a more or less deep red color appears.

$Fe^{++}$  )

Fe sol. p. 98

Dimethyl glyoxime

Limit 0.04  $\mu$  Fe

Concn. Limit 1,125,000

Reagents 1% alc. Dimethyl glyoxime

Tartaric acid

$NH_4OH$

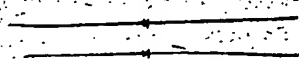
$Fe^{++}$  forms soluble red inner complex with



No this water is but if  $K_2Cr_2O_7$  added the  
 No salt dissolves & only the  $Fe^{2+}$  will remain  
 T. test for  $Fe^{2+}$  &  $Fe^{3+}$ , test with tartrate  
 → soluble complex stable in presence of  $NH_3$

method - Reduce  $Fe^{3+}$  with  $SO_2$  ( $H_2SO_5$ ) or  
 hydroxylamine hydrochloride

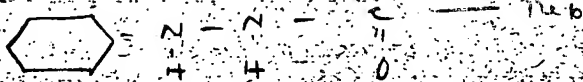
add a drop of the soln with a sol of  
 tartrate & then add a drop of a 5M/6  
 followed by a little  $NH_4OH$  according to  
 the  $Fe^{3+}$  content, a red color appears  
 red color appears which fades on stand-  
 ing due to redn of  $Fe^{3+}$  complex



Cr

millan p. 316

Reagents: Barium carbonate



and detectable 0.0002% mg Cr in a sol  
 of 1, 2, 10, 100

and determinable 0.015 to 0.05 mg Cr

interfering substances  $Co$ ,  $Cu$ ,  $Mn$  &  $Ni$

D.P.C. soln. 1% in EtOH

in water - (at aq. soln.)

Test tube - place a drop of HCl test soln  
on a plate & add 1 drop of 1% HCl  
then add 2 N NaOH dropwise until the  
resulting soln is alk to litmus. add  
a vol of phenol & 1 drop of 1% D.P.C.  
soln. & 2 N NaOH dropwise until the red  
color of alk-D.P.C. is dispersed. It disperses  
as purple then permanent violet blue color appears.

→

many times D.P.C. diss in 1% AcOH (al)  
& dil to 1% with 95% EtOH.

Qual. Test - To color of test soln add  
vol of 6 N  $\text{NH}_4\text{OH}$ , acidity to AcOH,  
add vol of D.P.C. reagent & shake.  
If color persists, violet or violet  
red color, indicate with 3% thio.

and detectable 0.001 mg/ml 1:1, 0.001, 0.001

interfering substances Al & Zn

Ca - Manganese - yellow n. 3.13 +  
( ) faint n. 1.52

Mn + vanadates interfere

Manganese soln - 1.150 in 714, Acid #1

NaVO soln - 1.145 (faint)

limit of ident - 1.251 in

2.500 - limit - 1.250, 1.000

reaction - place a drop of NaVO soln on a  
strip of filter paper, add a drop of  
this soln & a drop of Manganese soln  
H<sub>2</sub>CrO<sub>4</sub> is present, a blue ring appears.

M.O. - Methylhydrazine

soln detectable - 1.006 in concn of 1.100, 1.000

reaction - to 5 cc of test soln (containing methylhydrazine)  
add 5 cc of 2H soln (not 4H), 1.02 cc of  
HCl & heat  $\rightarrow$  red to blood red color

note - 1.14 in 2H soln - Acid soln - place a drop of  
test soln on spot plate & add a drop of  
2H reagent  $\rightarrow$  red color

detected 1.1404 in concn of 1.100, 1.000

No. KCN 1000 mg in 100 ml of 1% KCN  
Interfering substances Fe, Hg, Cu, Pb, As

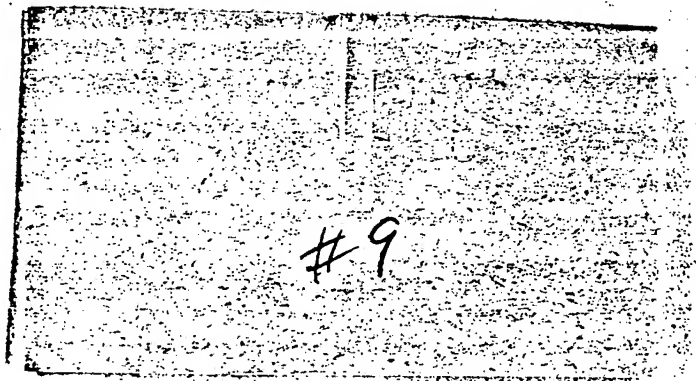
Dimethylglyoxime 10 ml 10% solution

Na 100 mg of Fe 1000 mg of Na (1000 mg of Na in 100 ml of 1% NaOH solution)  
Limit of Fe 0.5 mg Na 1000 mg  
Limit of Na 1000 mg

place on test plate

- 1 drop of 10% NaOH
- 1 drop of 10% NaOH
- 1 drop of 10% NaOH
- 1 drop of 10% NaOH

red Na not found at all & impure  
due to double C & E of Na



PROGRAM OF WORK FOR THE  
CHINESE JOB

8/26/46 and tempus fugit

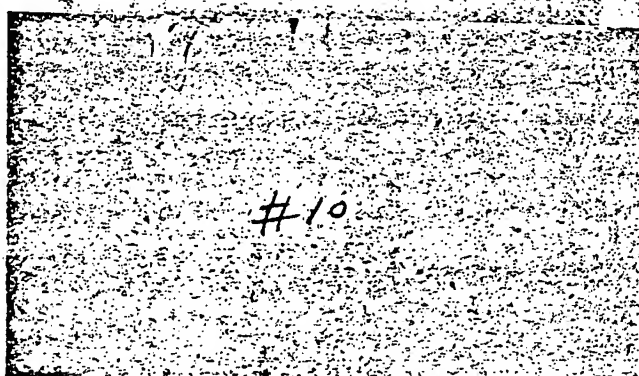
LAB SCHEDULE

- |                                                           |                   |
|-----------------------------------------------------------|-------------------|
| 1. Acetylene $\rightarrow$ Acetone                        | October 21, 1946  |
| 2. $\text{CO}_2$ and $\text{NH}_3 \rightarrow$ Urea       | November 4, 1946  |
| 3. Electrothermic Processes<br>a. Cyanamide<br>b. Cyanide | December 1, 1946  |
| 4. HCN Generation                                         | December 31, 1946 |
| 5. Acetylene Generation                                   | December 31, 1946 |

OFFICE SCHEDULE

- |                                   |                   |
|-----------------------------------|-------------------|
| 1. Preliminary Flowsheets         | Already completed |
| 2. Calculations                   | November 1, 1946  |
| 3. Instrumentation (preliminary)  | August 30, 1946   |
| 4. Unit Process Equipment         | November 30, 1946 |
| 5. Specified Equipment            | March 30, 1947    |
| 6. Piping, etc.                   | February 28, 1947 |
| 7. Power Distribution             | February 28, 1947 |
| 8. Steam and Water Distribution   | February 28, 1947 |
| 9. Building Design                | March 30, 1947    |
| 10. Power Plant                   | December 31, 1946 |
| 11. Layout and Placement Drawings | April 30, 1947    |
| 12. Lab. Facilities               | December 31, 1946 |





(Fe) as "iron"

2,2'-dipyridyl

limit 0.03% Fe

concentration 1: 1,666,000

2% in etc.

method - treat drop on spot plate → red color

(Cr) as  $CrO_4^{2-}$

2,2'-dipyridyl carbazide

limit 0.001 mg Cr

concentration 1: 1,000,000,000

dis. 1 min in 5%  $AcOH$

→ dil. to 100 cc with 45%  $NaOH$

method - 75% of the vol. add 2 cc of 6N  $NH_4Cl$

and by to  $AcOH$ , add 2 cc of 1% lead acetate & shake. If this appears → violet or violetish color.

(2) mixing with 5%  $H_2O_2$

(Mo) as molybdate

phenylhydrazine

limit 0.0004 mg

concentration 1: 100,000

add 10 cc of phenylhydrazine

in 100 cc  $AcOH$

method - place a drop on spot plate & add a drop of ph. reagent → red color

(Na) in presence of Fe

simultaneous reaction

limits 0.8 (1) - 4

continuous

1, 500, 000

0.470 in alc

method

1. do reaction

1. do test Na carbonate

2. do test NaCl

1. do test NaCl

→ Red Na test at edge of sample due to  
leakage of NaCl

0.5 - def. = 2.6 141

12.2112 am. orig

each 2000  
each 1000  
140

11.3039 am. orig

11.3033

11.3029

0.0005

W - 1000 → < 0.001 mag (1.1, 000, 000) - each 1000  
Fe - 1000 → 0.0006 mag (1.1, 000, 000) - each 1000  
Au - 1000 → 0.0006 mag (1.1, 000, 000) - each 1000

10.7742 am. orig

10.7742

10.7740

124-476

0.0000

10.7740

1100-476

0.0000

each 1000  
each 1000

7.4002 am. orig

7.4097

7.4092

0.0006

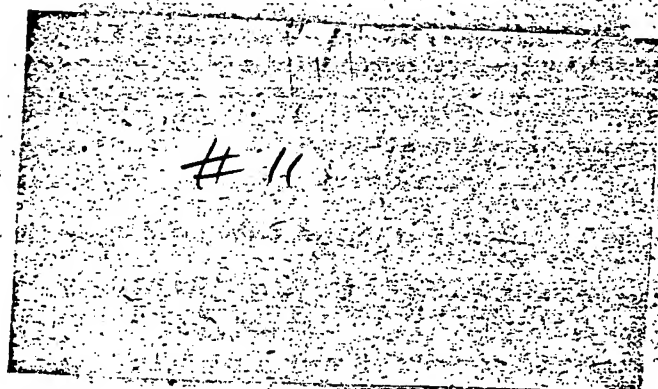
0.0006

7.4088

1100-476

each 1000  
each 1000

each 1000  
each 1000



Distillate of Acetic Acid

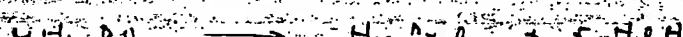
Found

1.44 gms, 2.00 gms  $H_2O$

4.00 gms residue

144.4 gms  $H_2O$  = 39.55 = 37.9, 144.4

14.4.0.0



4.61.3

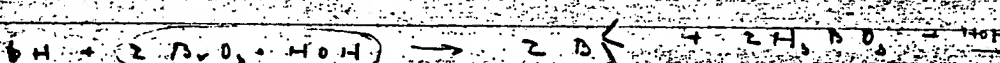
157.3

157.3  
4.61.3

152.7

78.7 gms

$H_2O$



1 mole water and 5 of acetic acid

78.7 x 0.97 = 68.5 gms actual  $H_2O$

Using only 78.7 gms

68.5 = 0.75 = 59 gms

|      |     |     |     |
|------|-----|-----|-----|
| 14.4 | 4.6 | 1.4 | 1.4 |
| 14.4 | 4.6 | 1.4 | 1.4 |
| 1    | 1   | 1   | 1   |
| 14.4 | 4.6 | 1.4 | 1.4 |
| 1    | 1   | 1   | 1   |
| 14.4 | 4.6 | 1.4 | 1.4 |
| 1    | 1   | 1   | 1   |
| 14.4 | 4.6 | 1.4 | 1.4 |
| 1    | 1   | 1   | 1   |

10.4 = 59.5 = 19.5 gms acetic acid

157.3



Sum of route of 204 0.3 and  
( )  
line 817.4 and 0.250 sum

195.000 = 5.9 sum.

Dehydration of AcOH via  $H_2SO_4$  - Toluene  
chem

17.6 gms. Toluene = 2 mols

1.0 gms. AcOH = 1 mol

9.1 gms.  $H_2SO_4$  = 1.5 mols

also 1.1 gms. added Toluene for 7 mols

distilled thru column to Midwell-Stillby trap

| Time            | Temp | Lower Layer | Notes |
|-----------------|------|-------------|-------|
| 7 $\frac{1}{2}$ | —    | 0           |       |
| 8               | 114  | 13          |       |
| 9               | 103  | 22          |       |
| 9 $\frac{1}{2}$ | 104  | 50          |       |

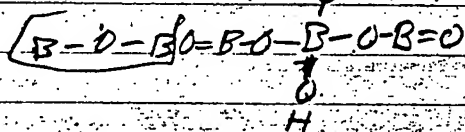
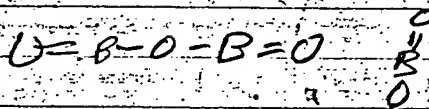
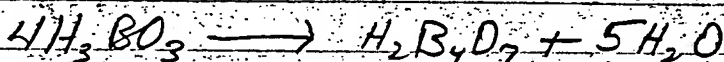
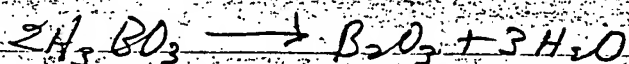
Retention  $\frac{40}{5} = 100$  ;  $\frac{114}{5} = 22.8$  ;  $\frac{103}{5} = 20.6$  ;  $\frac{104}{5} = 20.8$   
 out of 40

Final retention  $\frac{177}{5} = 35.4$  ;  $\frac{100}{5} = 20.0$  ;  $\frac{104}{5} = 20.8$   
 45 gms  
 AcOH

6.9  
 4.5  
 14.4 gms. 140.14

10.0 gms. 4.0  
 5.4  
 14.4 gms. 140.14  
 4.0 gms. 4.0  
 5.4  
 14.4 gms. 140.14

$$\left( \frac{5}{6} \times 3 = \frac{5}{2} = 2\frac{1}{2} \right)$$



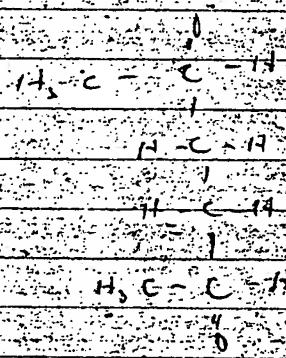
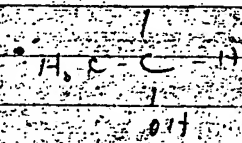
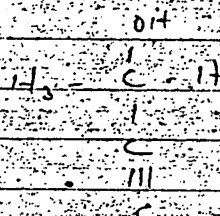
Hydriodide of antimony, dim  
( )

$$1000 \text{ cc} = 910 \text{ gms.}$$

$$9.32 \cdot \frac{132}{1.10} \cdot \frac{1}{1.185} = 1040 \text{ cc}$$

$$\text{Calc addn HCl} = 10 \frac{30}{\text{min}}$$

$$\text{Finn addn HCl} = \text{N.M.}$$



dist = 142 v.l. 14.2g  $\rightarrow$  0.2 mol  $\rightarrow$  3.6 cc

$$H_3BO_3 = 61.84 \quad \frac{0.24}{3} = \frac{12.1}{3} = 4.13g$$

$$109.6 \times 3 = 4.55$$

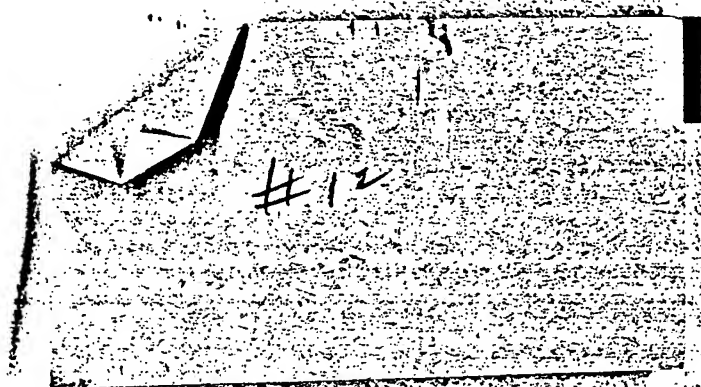
14.2g dist = 0.1M

5.58g  $H_3BO_3$  =

40 cc  $C_6H_6$

| Ref  | Column | HD  |
|------|--------|-----|
| 83   | 70°    | 0   |
| 6.45 | 77°    | 2.6 |





Cell characteristics  $1N H_2SO_4$  ca 1 minute anode (120cc. sol's)  
~~1.5~~  $I$  actual p.p.m.

|      |      |
|------|------|
| 3.3  | 1195 |
| 3.47 | 1232 |
| 3.68 | 1281 |
| 4.01 | 1493 |
| 4.52 | 1861 |

Cell characteristics  $1N H_2SO_4 + 1.43g$  diol

$E$   $I$   $E$   $I$

|      |             |                             |                 |
|------|-------------|-----------------------------|-----------------|
| 3.0  | 1215 (1195) | 3.51                        | 1272            |
| 3.32 | 1215        |                             |                 |
| 3.4  | 1226        | 3.51 with 5/12              |                 |
| 3.46 | 1242        | 11.30 - 361 Amp             |                 |
| 3.57 | 1273        | 12.00 Reduced 5.18 + 2.50   |                 |
| 3.89 | 1445        | At 12.00 Reduced 5.33 + 1.8 |                 |
| 3.71 | 1400        | at 8"                       | (5.50) or 0.170 |
| 3.66 | 1363        |                             | 3.27            |
| 3.63 | 1352        | 3.28                        | 1181            |
| 3.61 | 1385        | 3.38                        | 1208            |
| 3.60 | 1323        | 3.45                        | 1221            |
| 3.58 | 1311        | 3.55                        | 1296            |
| 3.57 | 1300        | 3.60                        | 1260            |
| 3.55 | 1290        | Revised 1.28 g              |                 |
| 3.52 | 128         |                             |                 |

221.42.00  
118

22

Ni bedrock -  $\pi d = 3.14 \times 2 = 21.98$  Depth of 4.55 m. = 1 Diameter

$\pi r^2 = 38.5 \text{ sqm}$   $38.5 \times 4.55 = 175 \text{ cc}$  (165)

Cell cont'g. 165 cc. RN NaOH outside cup

Voltage of cell  $\frac{\text{area}}{\pi d}$  ca. 0.3 volt - exp. falling

E I

2.30 .105

2.39 .153

2.51 .236

2.62 .325

2.78 .427

2.95 .595

3.19 .840

3.35 .105

Added 4.26.00

3.34 .105

2.44 .152

2.59 .233

2.70 .311

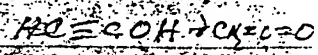
2.79 .379

$\begin{array}{r} 241.66 \\ 3.04 \end{array}$ 
 $\begin{array}{r} 64 \\ 1.61 \\ 9.66 \text{ avg hr.} \end{array}$ 
 $\begin{array}{r} 84 \\ 84 \end{array}$ 
 $\begin{array}{r} .06 \\ 6615 \end{array}$

75cc. 1N  $\text{H}_2\text{SO}_4$  + 5g butanol outside porous cup.

35 - 1st reading

| F    | T          | E    | I      |
|------|------------|------|--------|
| 2.12 | 1.202      | 2.99 | .087   |
| 3.06 | 1.165      | 3.05 | .120 ✓ |
| 3.16 | 1.203      | 3.20 | .134   |
| 3.20 | 1.220      | 3.23 | .130   |
| 3.23 | 1.233      | 3.29 | .125   |
| 3.28 | 1.2405     | 3.25 | .160   |
| 3.32 | 1.252      | 3.22 | .155   |
| 3.29 | 1.262      | 3.19 | .140   |
| 3.94 | 1.277      | 3.16 | .123   |
| 3.50 | 1.304      | 3.12 | .111 ✓ |
| 3.59 | 1.335      | 3.08 | .102   |
| 3.64 | 1.360      | 3.10 | .1203  |
| 3.75 | 1.407      | 3.50 | .136 ✓ |
| 3.83 | 1.448      | 3.44 | .1219  |
| 3.95 | 1.499 1.15 | 3.45 | .1220  |
| 3.93 | 1.082      | 4.24 | .1592  |
| 07   | 1.120 4.5  |      |        |
| 50   | 1.730 1.85 |      |        |
| 120  | 1.175 6.35 |      |        |



Sunday. 2/29/48  
Shred 21.3 g. glycol with 112 cc conc HCl 2 hrs.  
Sep & washed.

|    | lip.      | $n_D^{21.5}$ | $n_D^{23}$ |
|----|-----------|--------------|------------|
| 1. | 66-72/14m | 1.4285       | 1.4778     |
| 2. | 72-76/15m | 1.4800       | 6.8g.      |
| 3. | 76-8/15m  | 1.4939 (21)  | 6.6g.      |

$$\begin{array}{r} 12659 \\ 2525 \\ \hline 15184 \\ 420 \end{array}$$

$$\begin{array}{r} 16405 \\ 15184 \\ \hline \end{array}$$

$$\begin{array}{r} 1221 \\ \hline \end{array}$$

$$\begin{array}{r} 12435 \\ 60 \\ \hline \end{array}$$

$$\begin{array}{r} 366 \\ 4255 \\ \hline 455 \end{array}$$

$$\begin{array}{r} 1424 \\ 72 \\ \hline 362 \\ 432 \end{array}$$

$$420$$

$$\begin{array}{r} 1287 \\ 2005 \\ \hline 544 \\ 3839 \end{array}$$

$$\begin{array}{r} 12396 \\ 2525 \\ \hline \end{array}$$

$$\begin{array}{r} 14861 \\ \hline \end{array}$$

$$\begin{array}{r} 16465 \\ \hline \end{array}$$

$$\begin{array}{r} 14861 \\ \hline \end{array}$$

$$\begin{array}{r} 1544 \\ \hline \end{array}$$

$$1544 \times 35$$

$$75$$

$$\begin{array}{r} 45 \\ \hline \end{array}$$

$$\begin{array}{r} 525 \\ \hline \end{array}$$

$$\begin{array}{r} 540 \\ \hline \end{array}$$





1116.5

114

$\text{ClCH}_2\text{COONa}$  1690 lb

$\text{ClCH}_2\text{COONa}$  1690 lb

$\text{ClCH}_2\text{COOH}$  150 lb

$\text{H}_2\text{O}$  4253

$\text{CO}_2$  8.26

$\text{H}_2\text{S}$  67.4

$\text{ClCH}_2\text{COONa}$  3.13

$\text{NaHS}$  273

$\text{HSCH}_2\text{COONa}$  302

$\text{NaCl}$  153.7

$\text{HSCH}_2\text{COOH}$  { 223

$\text{NaOH}$  { 228

$\text{NaCl}$  { 153

$\text{H}_2\text{SO}_4$  { 513

$\text{H}_2\text{O}$  { 1729

$\text{H}_2\text{S}$  67.4

$\text{ClCH}_2\text{COONa}$  31.7

$\text{NaHS}$  41

$\text{HSCH}_2\text{COONa}$  274

$\text{NaCl}$  139.5

$\text{H}_2\text{O}$  1612

2.2N thioglycolic acid = 198.5 g/lit

$\text{H}_2\text{S}$  16.85  
 $\text{ClCH}_2\text{COONa}$  7.98 - 5.83  $\text{ClCH}_2\text{COOH}$  + 3.27g  $\text{Na}_2\text{CO}_3$   
 $\text{ClCH}_2\text{COONa}$  68.5 - 55.3g  $\text{HSClCH}_2\text{COOH}$  + 25.4g  $\text{NaOH}$  28  
 $\text{NaHS}$  10.3 - 7.75g  $\text{NaOH}$   
 $\text{NaCl}$  34.88  
 $\text{H}_2\text{O}$  403  
 $\text{NaHS}$  3

$\text{HSClCH}_2\text{COOH}$  56 38  
 $\text{Na}_2\text{SO}_4$  56 28  
 $\text{NaCl}$  38 19  
 $\text{H}_2\text{SO}_4$  1.3 0.65  
 $\text{H}_2\text{O}$  432 218  
 12.5g

Extract 530 cc,  
 once with equal vol. butyl ether

$\text{ClCH}_2\text{COONa}$  15 {13g} = 153g  $\text{ClCH}_2\text{COOH}$  + 77.5g  $\text{Na}_2\text{CO}_3$   
 $\text{ClCH}_2\text{COOH}$  15 {15g}  
 $\text{H}_2\text{O}$  425.3  
 500  
 76.5g  $\text{ClCH}_2\text{COOH}$  + 38.8g  $\text{Na}_2\text{CO}_3$   
 202.7 cc  $\text{H}_2\text{O}$

$\text{H}_2\text{S}$  8.43  
 $\text{ClCH}_2\text{COOH}$  2.92  
 $\text{Na}_2\text{CO}_3$  1.64  
 $\text{HSClCH}_2\text{COOH}$  27.7  
 $\text{NaOH}$  12.7  
 $\text{NaOH for NaHS}$  3.87  
 $\text{NaCl}$  17.44  
 $\text{H}_2\text{O}$  201.5 cc  
 Extract 530 cc, once with equal vol. butyl ether